WANL-PR-M-014
October 30, 1965

Westinghouse Astronuclear Laboratory

Pilot Production And Evaluation Of Tantalum Alloy Sheet

Prepared by:
R.L. Ammon
A.M. Filippi
D. L. Harrod

Prepared Under Navy
Contract NOw-64-0394-d

SUMMARY PHASE REPORT
PART III

April 28, 1964 to October 30, 1965

Astronuclear Laboratory

UNCLASSIFIED

WANL-PR-M-014
October 30, 1965

PILOT PRODUCTION AND EVALUATION OF TANTALUM ALLOY SHEET

Prepared by R. L. Ammon, A. M. Filippi and D. L. Harrod

Summary Phase Report
Part III

Prepared Under Bureau of Naval Weapons
Contract NOw-64-0394-d

April 28, 1964 to October 30, 1965

Astronuclear Laboratory
Westinghouse Electric Corporation

UNCLASSIFIED

ABSTRACT

Two 80-pound, 4-inch diameter double arc cast T-222 ingots were produced and converted to three wide sheets. Excellent primary (forging) and secondary (rolling) fabricability were displayed by the material, and an approximately 1/3 ingot-to-sheet conversion yield was obtained. The sheets displayed physical and mechanical properties comparable to those previously reported for T-222, as well as good product consistancy.

Phase identification studies, which were begun on a previous contract, (N600(19)-59762), were continued and broadened during this work phase. Light microscopy, electron microscopy, and x-ray diffraction techniques were employed to study the morphology and structure of precipitated phases in T-222. Second phases present after aging T-222 were found to be dependent upon mechanical-thermal history. For example, aging as-rolled material at temperatures from 1600 to 2000°F produced the FCC monocarbide phase, (Hf,Ta,W)C, while in contrast aging solution treated material in this temperature range produced the HCP dimetal carbide phase only, $(Ta,W,Hf)_2C$. Preferential grain boundary nucleation or phase stabilization due to dissolved oxygen or nitrogen are thought to be probable explanations for the anomolous behavior.

Analysis of metallographic and mechanical property data on T-222 indicate that the interstitial strengthening effect may result from immobilization of dislocations by strain induced precipitation and/or precipitation during cooling from the solution annealing temperature. Results of this study also indicate that the optimum metallurgical condition for T-222 alloy is that obtained by recrystallization and solution annealing. In general, a one hour anneal at 3000°F will provide this condition.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

I. INTRODUCTION

This is the final report on the "Pilot Production and Evaluation of Tantalum Alloy Sheet" program sponsored by the Bureau of Naval Weapons under Contract NOw-64-0394-d. The program was a continuation of previous studies conducted under Contracts NOw-62-0656-d and N600(19)-59762, the results of which are summarized in References 1 and 2. This report covers the work period April 28, 1964 through October 30, 1965.

The objectives of the program were as follows:

1. Investigate the mechanical properties of T-222 sheet as a function of prior mechanical-thermal history to determine if mechanical properties could be further optimized by modifications of the standard process developed for the conversion of T-111 and T-222 arc cast ingot to sheet.
2. Continue investigation of the precipitation reactions in T-222 with the purpose of correlating mechanical properties with observed microstructural features.
3. Produce wide (approximately 24 inches) T-222 sheet from 4-inch diameter arc cast ingots using a process which incorporates the findings of objectives (1) and (2) above.

Item (3) constituted the major effort of the current program. In meeting this objective, two, 4 inch diameter T-222 ingots, each weighing approximately 80 pounds, were double arc melted and converted to 0.040 inch thick sheet. To evaluate fabricability both upset and side forging were investigated as ingot breakdown techniques.

Stress-rupture, high temperature tensile, recrystallization, phase identification, and aging response data were acquired on samples from one sheet, while check chemical analyses, room temperature tensile, weld bend and sheet bend data were obtained in all three sheets. Flatness of each sheet was also determined in accordance with MAB-184-M.

The response to heat treatment of this wide sheet was studied and compared to results obtaine on sheet from two smaller T-222 heats, one of similar carbon content (about 100 ppm), and one of much higher carbon content (400 ppm). Comparison of the second phases present after aging at temperatures from 1600 to 4000°F were made on samples extracted from these heats. In addition to this, bulk samples were examined by light and electron microscopy to characterize the structures developed.

Phase identification studies were completed, and the correlation between observed microstructural features and mechanical properties was established. Studies of the effect of thermomechanical treatments on the properties of T-222 indicated that a 1 hour anneal at 3000°F provided the optimum combination of strength and ductility.

II. GENERAL EXPERIMENTAL PROCEDURE

HEAT TREATING

All material for heat treatment was degreased, etched in a HNO_3-H_2SO_4-HF-H_2O solution, and wrapped in clean tantalum foil. All treatments were carried out in vacuum at pressures of 5×10^{-5} torr or below. Temperature control was maintained by means of calibrated micro-optical pyrometer readings of a black body sample within the hot zone with appropriate corrections for transmittance and absorption of the sight glass.

MECHANICAL TESTING

The gauge section of the tensile and stress-rupture bars was 1 inch by 0.25 inch by sheet thickness. Blanks of the appropriate size were cut from sheet stock, surface ground, etched, and heat treated. Test bars were then machined from the blanks, cleaned, and tested. For high temperature tests, clean tantalum foil was wrapped loosely around the gauge section. High temperature tensile tests were conducted at Metcut Research Associated, Cincinnati, Ohio. Test conditions such as strain rate, temperature control and measurement, heating rate, and test environment recommended by the MAB were followed.[3]

Stress-rupture tests were carried out at the Westinghouse Astronuclear Laboratory in two Satec VC-100 vacuum creep machines. Pressure during testing was maintained below 5×10^{-5} torr. The gauge sections were also wrapped with tantalum foil prior to testing. For test temperatures from 2000°F to 3000°F three Pt/Pt-Rh thermocouples were attached to the gauge section, one at each end and the control couple at the center of the gauge section. The strain-time curves were taken using a Satec extensometer in conjunction with a Wiedemann measuring system.

Low temperature tensile tests were conducted in a hard tensile machine at a constant strain rate of 0.05 in/min.

Room temperature hardness measurements were made using a Vickers hardness machine with a 10 kilogram load.

WELDING

Sheet TIG butt welds were made in a vacuum purged weld box backfilled with high purity helium. The sheet stock was rigidly held in a copper and stainless steel clamping device designed to give high restraint while providing a heat sink for rapid cooling of the welded sheet. Sheet, 0.040 inch thick, was welded at a current of 95 amps, an arc gap of 0.062 inch, with a travel speed of 7.5 inches per minute.

Bend tests were conducted using specimens 12T wide by 24T long with the weld parallel to the long dimension. The span was fixed at 15T and deflection rate was 1 inch per minute. Specimens were tested with the top of the weld in tension and were bent through an angle of greater than 90 degrees or until a sudden drop in load was observed, at which time the test was stopped. Bend angles were measured in the unloaded condition. Test temperatures from -200 to -320°F were obtained by regulating a flow of gaseous nitrogen (produced from liquid nitrogen) onto the specimen. Temperatures were read from thermocouples attached at the punch and specimen button which in turn, through suitable solonoid valves, regulate the coolant flow.

METALLOGRAPHY

The samples, mounted in one and one-quarter inch diameter Bakelite mounts, were ground and polished on Buehler Automets. Five mounts were done simultaneously in a specimen holder.

Grinding was done at 40-pounds pressure on 240, 400, and 600 grit water cooled papers. The samples, still in a specimen holder, were then hand ground on three-zero and four-zero emery paper. The preliminary polishing was done with diamond compounds 30 micron, 15 micron, and 6 micron on Buehler Metcloth at 30 pounds pressure.

Using dry Linde A polishing abrasive, an intermediate acid polish containing a mixture of 100 milliliters of acetic acid, 60 milliliters of nitric acid, and 3 milliliters of hydrofluoric acid, was used on micro cloth applying 30 pounds pressure for eight minutes. The final polish, using 10 pounds pressure for one minute, was performed using the same procedure as the intermediate polish.

The samples were swab erched with a solution of 50 milliliters nitric acid, 30 grams ammonium bifluoride and 20 milliliters water.

TABLE 1 - Analysis of Starting Material

Material	Supplier	Form	Analysis (ppm)																
			C	O	N	Al	Cb	Cr	Fe	Mn	Mo	Ni	Si	Ta	Ti	V	W	Zr	Ash
Ta-10W	Fansteel	Plate	10	10	27	10	70	10	10	10	60	10	30	Bal.	10	10	9.9*	10	--
Hf	Carborundum	Plate	80	360	24	20	50	5	34	5	10	10	20	200	20	5	--	1.7*	--
W	Wah Chang	Sheet	4	300	--	10	50	10	20	--	--	20	10	50	--	--	Bal.	--	--
C	Union Carbide	Cloth	Bal.	--	---	--	--	--	--	--	--	--	--	--	--	--	--	--	400

*Per cent

III. PRODUCTION OF WIDE T-222 SHEET

Sheet material for the program was produced from two double vacuum arc cast T-222 ingots nominally 4 inches in diameter. First melts were made into a 2-3/8 inch mold using A. C. power, while D. C. power was used for the second melt. Sheet was produced by cold rolling conditioned and recrystallized sheet bar obtained by upset forging or side forging the as-cast ingot.

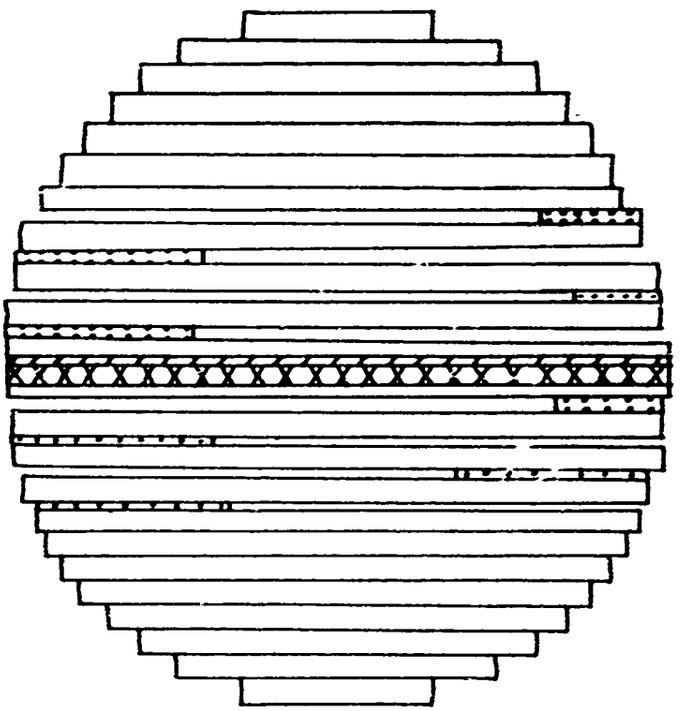
MELTING

Starting electrodes were assembled basically as a sandwich of hafnium, tantalum-10 w/o tungsten and tungsten strips. Carbon was added as cloth wrapped in tantalum foil and inserted at the center of the sandwich. Chemistry of starting materials is given in Table 1.

Figure 1 shows the cross-section of a first melt electrode. The components as shown assembled in this figure provided the basic T-222 composition over the cross-section at any point along the electrode length. Each electrode weighed 40 pounds, and was 1-3/4 inches in diameter by 29-7/8 inches long. Fusion welds were made around the electrode periphery at intervals of 4 inches along the length to hold the bundle together for melting.

First melts were made into 2-3/8 inch diameter mold using A. C. power at 2800 to 3300 amps and 26 to 30 volts. Two melts had to be made per electrode since the length of the first melt mold limited the ingot weight to 20 pounds. Approximately 2 inches of the top of each electrode was also rewelded onto the next electrode to be melted. In this manner, the only material losses encountered in first melting resulted from saw cuts, and close to 100% electrode-to-first melt yield was obtained.

Two second melt electrodes weighing approximately 80 pounds were assembled from the first melt ingots by studding and butt welding. Four first melt ingots were joined in this manner for each second melt electrode.



Ta-10 w/o W

W

Hf

C cloth wrapped in Ta foil

SCALE: 1" = 1/2"

FIGURE 1 - Cross Section of First Melt T-222 Electrode

In order to be satisfied that the available electrical power for melting would be sufficient to allow production of a 4 inch ingot, attempts were made to melt a scrap pressed-and-sintered-powder tantalum base electrode into a 4 inch mold. Although this electrode was successfully melted and produced a satisfactory 4 inch ingot, the maximum available D. C. power (210 KW) had to be used. Because of this, it was felt that only marginal power was available for melting 4 inch ingot at Westinghouse, and the decision was made to make the second melts at the Bridgeville, Pennsylvania plant of the Universal Cyclops Corporation.

Two second melt ingots weighing 80 pounds were successfully produced at Universal Cyclops. The melts were made into a 4-1/2 inch diameter mold (a 4 inch diameter mold was not available). As it turned out only 175 to 190 KW of D. C. power was required to produce satisfactory ingot. The attempt to melt the scrap powder electrode into a 4 inch mold at Westinghouse undoubtedly required a higher power because of its high resistivity.

Nominal first and second melt data are shown in Table 2, and a photograph of a second melt ingot in Figure 2. The heat number marked on the ingot top shown in Figure 2 is that given it by Universal Cyclops. Heat numbers KC1452 and KC1455 were used by the Cyclops Corporation to distinguish between these two ingots, however, since they were both produced from the same starting material under identical melt conditions Westinghouse considers both to be of one heat. Westinghouse heat number Ta-43, which logically follows the successive T-222 melt identification system established under prior contracts,[2] therefore, more properly identifies the material. The Universal Cyclops heat numbers will be used from here on only to distinguish the two ingots apart. Photographs of the macrostructure at top and bottom of ingot KC-1452 are displayed in Figure 3. A relatively fine grain structure was attained with very little evidence of a columnar solidification pattern. Samples to check the major alloying element chemistry were removed from top and bottom of each ingot. Results are listed in Table 3.

TABLE 2 - Nominal First and Second Melt Data

Electrode Diameter (inches)	Melt Diameter (inches)	Melt Voltage (volts)	Melt Power (KW)	Melt Rate (lb/min.)
1-3/4*	2-3/8	26/30	80/100	4.5/5.5
2-3/8**	4-1/2	33/35	175/190	3.3/3.5

* A. C. Power
** D. C. Power

TABLE 3 - Analytical Results from Second Melt Ingots*

		Analysis (w/o)		
Heat No.	Sample	W	Hf	C
Ta-43 (KC1452)	Top	10.0	2.58	0.0090
	Bottom	10.6	2.71	0.0117
Ta-43 (KC1455)	Top	10.5	2.71	0.0092
	Bottom	10.7	2.66	0.0097

* The recommended specification range of T-222 is listed in Appendix A

FIGURE 2 - Photograph of T-222 second melt ingot. Flow of molten metal to the mold wall was adequate. The dark area at the ingot bottom is the pad which was used for the arc strike. T-222 chips, partially fused together, were used for the pad.

FIGURE 3 - Macrostructure at top and bottom of T-222 second melt ingot. Number KC-1452 (Heat Ta-43) 2/3X

FORGING

Primary breakdown from ingot to slab was accomplished by forging on a Model 1220C Dynapak at 2600°F. The method used to preset the proper energy for forging on this unit has been described previously.[2] Ingot fabricability was judged by comparing the condition of slab produced by both upset and side forging. Both ingots were lathe turned to 4-1/4 inches in diameter and locally conditioned by repeatedly hand grinding and etching, followed by liquid penetrant inspection until all surface flaws were removed. The conditioned ingot diameter averaged 4-3/16 inches. To minimize oxidation during forging billets were hot dip coated with an Al-12 w/o Si alloy. Billets were brought to forging temperature by induction heating in an atmosphere of flowing argon.

Ingot KC-1452 was cut into two lengths of 3-1/8 inches and 3-11/16 inches. The 3-1/8 inch length was upset forged to 1.15 inch slab, a 2.8/1 upset ratio, by two Dynapak forging blows utilizing a total of 4.5 million inch-pounds of energy. The 3-11/16 inch length was side forged, also using two Dynapak blows, to 1.5 inch thick slab; a diametrical upset of 2.8/1.

Photographs of both sides of the resulting slabs are displayed in Figures 4 and 5. A slight bit of tearing, probably due to anvil friction, can be noted on the upset forge produced slab, while the side forged piece is essentially tear-free. Some metal folding can also be noted around the edges of the upset piece, which is normally a result of this type of primary fabrication, especially as reduction ratios approach 3/1. In general, the surface condition of the side forge produced slab was somewhat better than the upset produced slab, but the differences were very slight, and the results of both primary breakdown procedures serve to display the excellent fabricability of T-222.

Although both the upset and side forge procedures resulted in a satisfactory slab for rolling to sheet, it was decided that side forging the remaining ingot (KC-1455) would produce a more satisfactory yield since it: (1) eliminates cutting the ingot into the short lengths necessary for upset forging; (2) produces a more rectangular shape as compared to upset produced slab, thus minimizing material loss on trimming to yield a rectangular sheet bar. Because of these

FIGURE 4 - Photograph of Upset Forged T-222 Section from Ingot KC-1452 (Heat Ta-43)

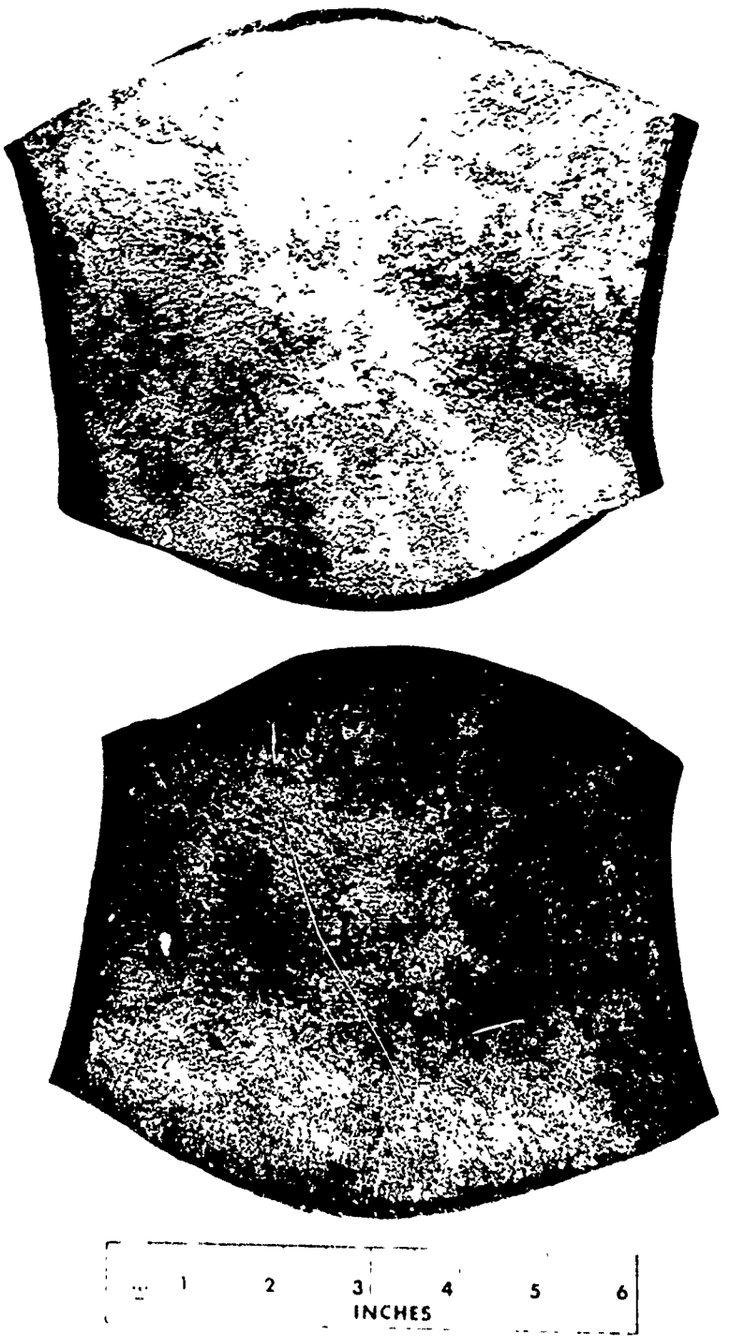


FIGURE 5 - Photograph of Side Forged T-222 Section from Ingot KC-1452 (Heat Ta-43)

considerations, ingot number KC-1455 was side forged in its entirety. This was done in a manner similar to that described above for the smaller pieces except that four Dynapak blows were utilized. The conditioned and coated ingot weighed 62 pounds and was forged to 1-11/16 inch thick slab. Calculations indicated that the four Dynapak blows would produce a 1-1/2 inch thick slab, but loss of heat during the forging cycles resulted in a somewhat thicker piece. Photographs of both sides of the resulting slab are shown in Figure 6. It is evident from these photographs that excellent quality slab was achieved.

ROLLING

The upset, and small and large side forged slabs, were trimmed to sheet bars weighing 14.3, 19.2, and 39.9 pounds, respectively. All three sheet bars were recrystallized at 2700°F for 2 hours, and cross-rolled to wide sheet. The 14.3 and 19.2 pound sheet bars were warm rolled (500 to 700°F) to a 18 inch length, re-annealed for 2 hours at 2700°F, and cross-rolled to 0.040 inch thickness. The 39.9 pound sheet bar was warm rolled to a 24 inch length, annealed, and cross-rolled to 0.040 inch thick sheet. An ingot-to-sheet flow chart is presented in Figure 7. To distinguish between the three sheets they have been identified Ta-43-U.F.-1, Ta-43-S.F.-1, and Ta-43-S.F.-2, relating to sheet produced from the upset forging, the small side forging, and the large side forging, respectively. It should be noted that the ingot-to-sheet yield was approximately 35% for all three pieces. The fact that the yield of 24 inch wide sheet, was as good as that of the smaller 18 inch sheet was undoubtedly due to the better bar yield resulting from side forging the entire ingot. Photographs of two of the sheets are shown in Figures 8 and 9.

IV. EVALUATION

FLATNESS TOLERANCE

The flatness tolerance was determined for all three sheets in accordance with paragraph 7e of Materials Advisory Board Specification MAB-184-M. This tolerance is reported as a percent flatness, and is determined by the maximum height of a bow divided by the minimum distance from the crest to a point of sheet contact with a flat surface. The flatness tolerance

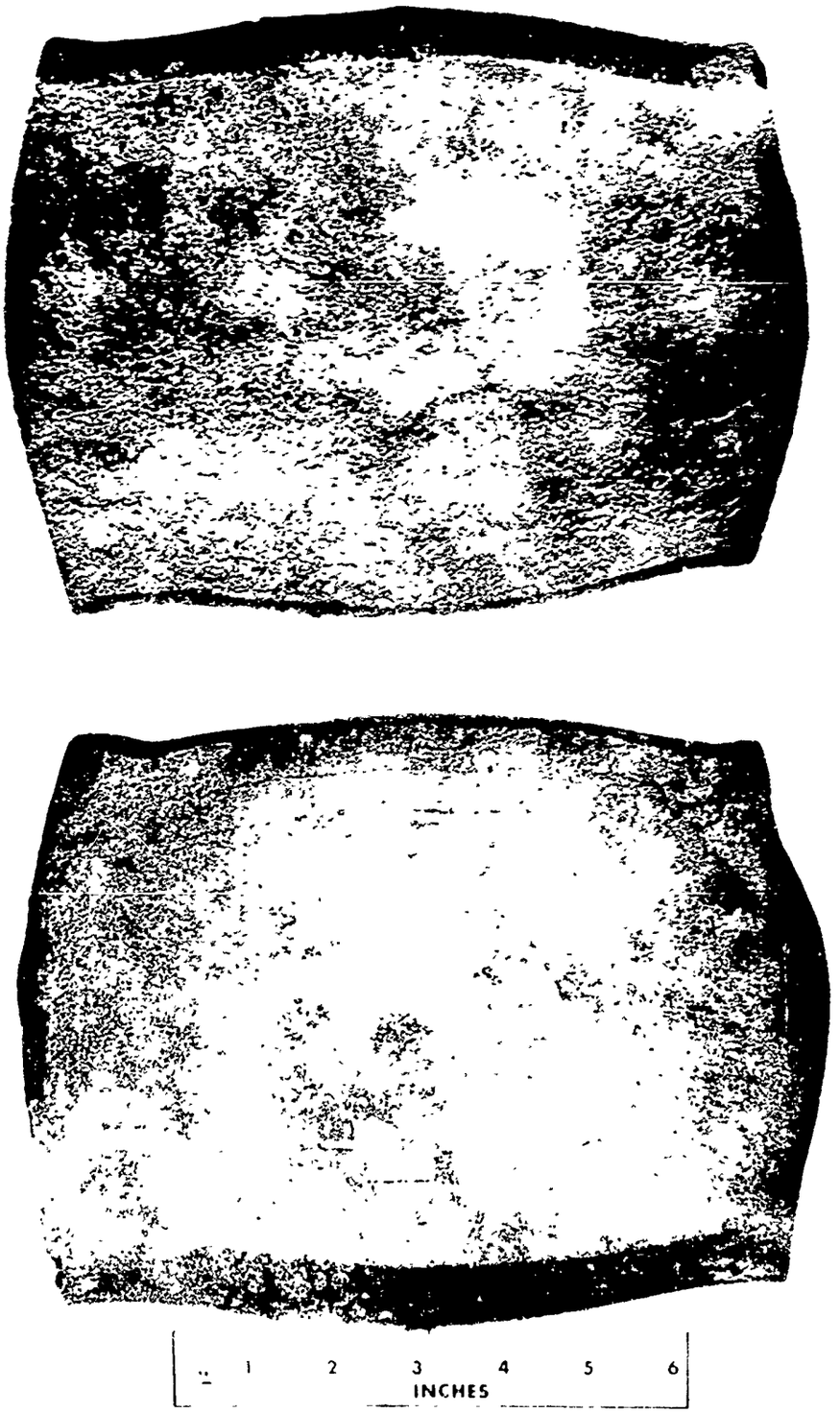


FIGURE 6 - Photograph of Side Forged T-222 Ingot KC-1455 (Heat Ta-43)

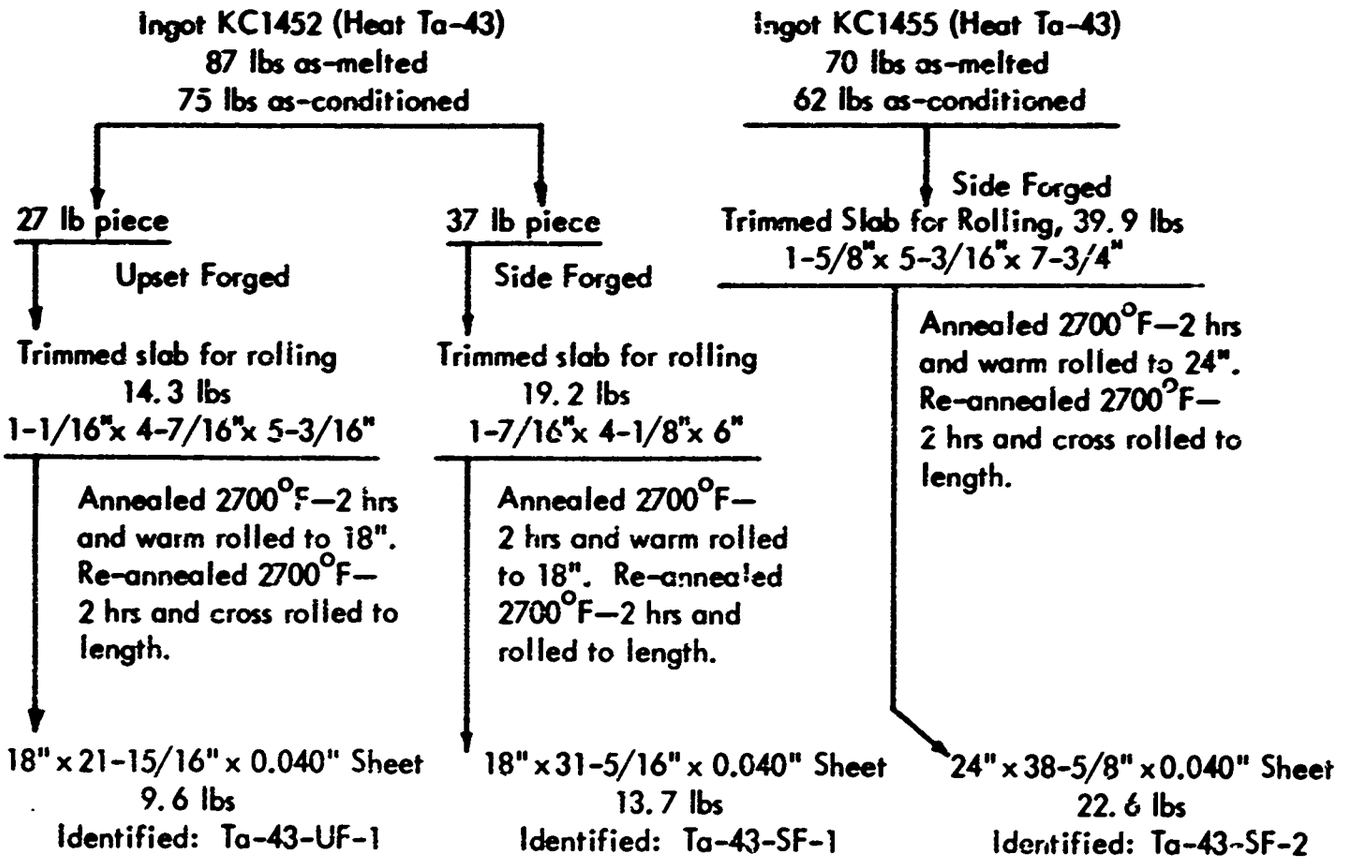


FIGURE 7 - Flow Chart of T-222 Ingot-to-Sheet Procedure

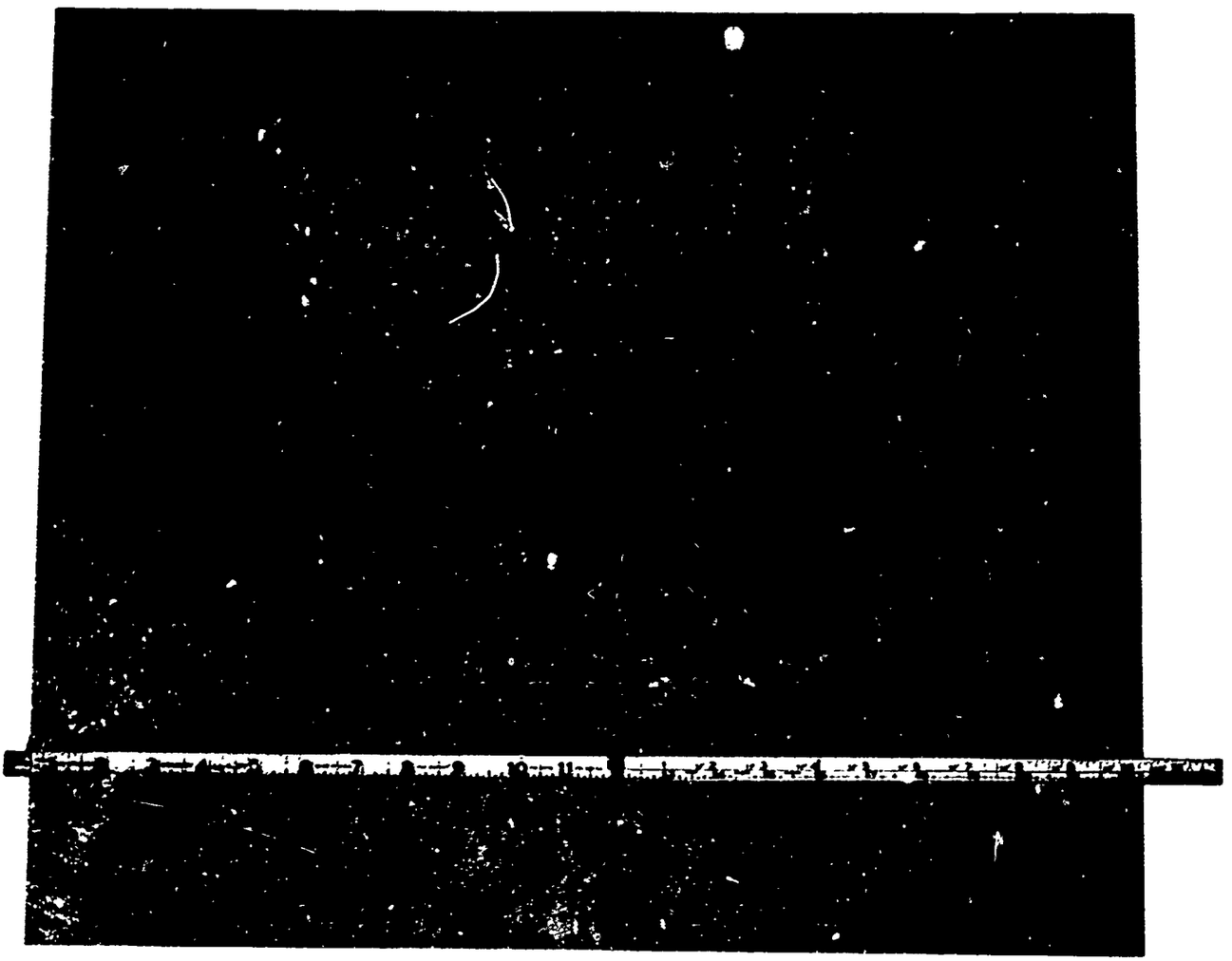


FIGURE 8 - Photograph of T-222 Sheet Ta-43-UF-1

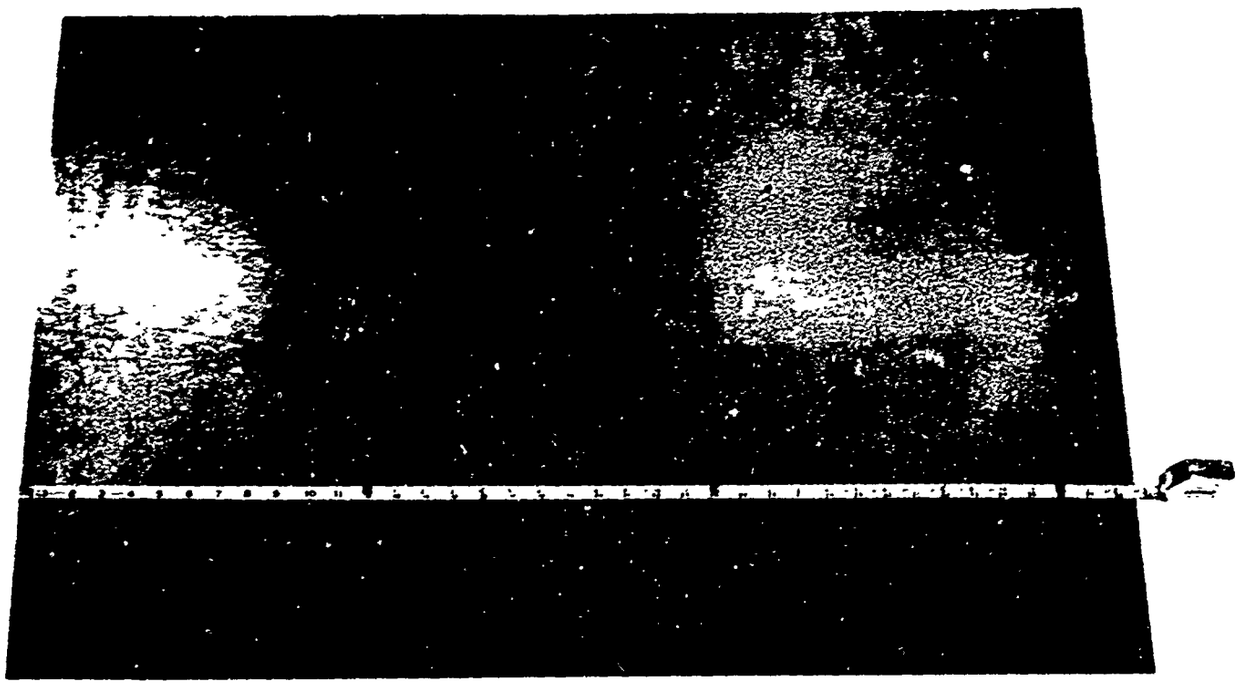


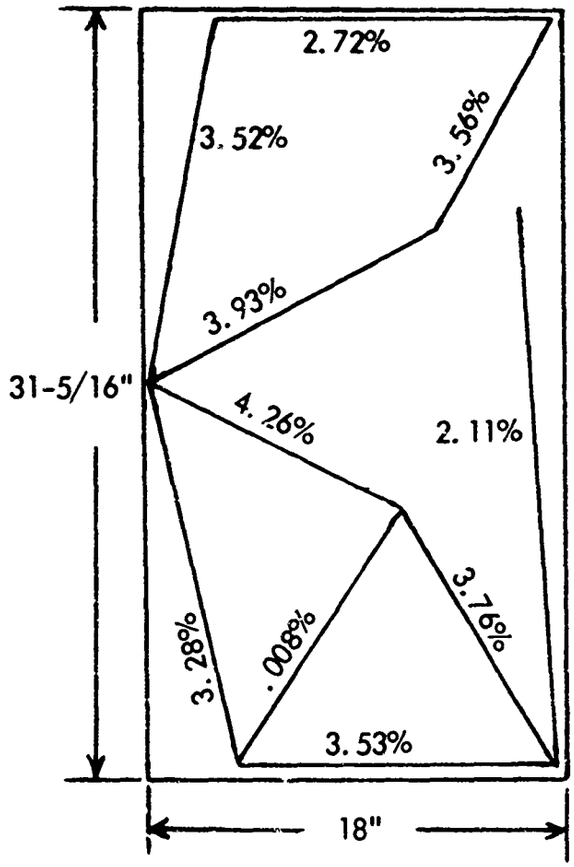
FIGURE 9 - Photograph of T-222 Sheet Ta-43-SF-2

on acceptable sheet should not exceed 4%. Figures 10, 11, and 12 display scaled layouts of each sheet including a description of their flatness. It is observed that although the sheets all contain many bows, on only one, S. F. -1, is the 4% tolerance maximum exceeded. Better control of sheet flatness could have been achieved if a larger number of sheets were processed to provide more experience in rolling wide T-222 sheet.

CHEMICAL ANALYSES AND MECHANICAL PROPERTIES

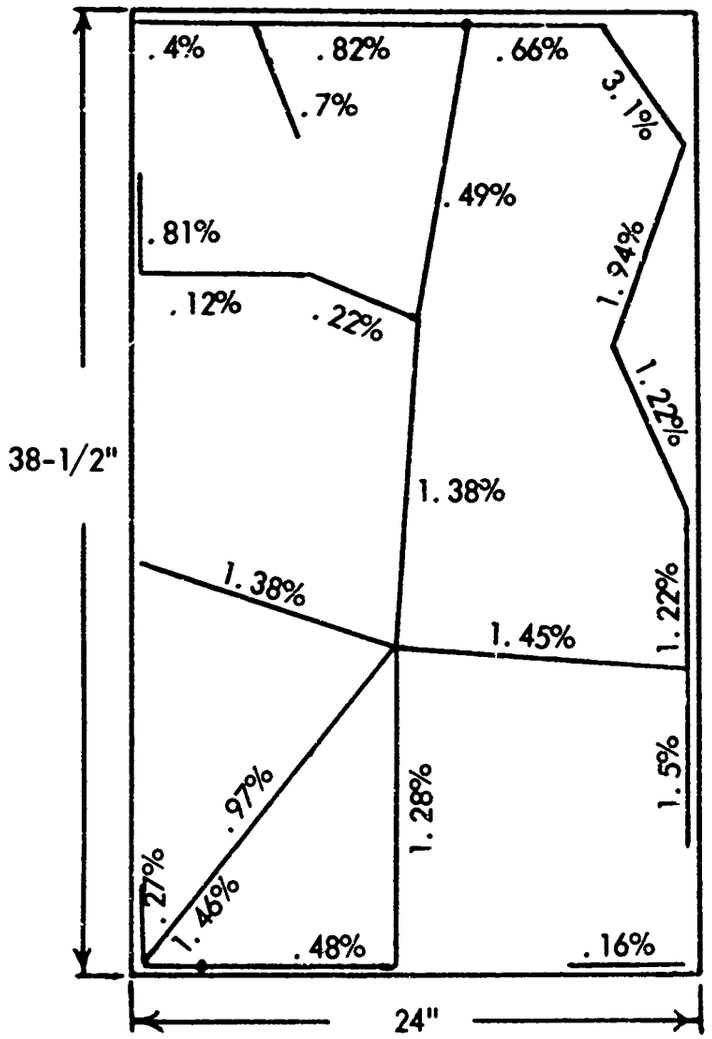
To evaluate product consistency, room temperature tensile tests, base metal and weld bend tests, and chemical analyses were obtained on samples from all three sheets. Transverse tensile tests were employed following the recommendations of MAB-184-M. Some room temperature tensile tests on longitudinal specimens were also carried out to provide a better comparison with results from previous heats. The longitudinal tests were only carried out on sheet Ta-43-S. F. -1. Elevated temperature tensile and stress-rupture properties as well as recrystallization characteristics were also evaluated using material from sheet Ta-43-S. F. -1. All mechanical property specimen were recrystallized by annealing 1 hour at 3000°F prior to test to permit a direct comparison with previous results on T-222.[2] Weld samples were annealed before welding, and tested without post weld heat treatment.

Analytical results are listed in Table 4. A sample from sheet Ta-43-S. F. -1 displayed a rather high copper content (60 ppm), and because of this a second sample was analyzed. It was thought that a high copper content would be reflected in poor elevated temperature properties and, therefore, analytical material for a second sample was removed from a stress-rupture specimen which displayed questionable results. Only 10 ppm copper was analyzed in this sample which is too low to be of any consequence, and is in line with the results on the other sheets. The elevated temperature tensile and stress-rupture data on sheet Ta-43-S. F. -1, to be discussed subsequently, deviated, but not markedly, from previously reported T-222 data, and further implied that the original high copper analysis was in error. Without qualification the remaining chemical analysis data summarized in Table 4 are in the range expected for T-222.



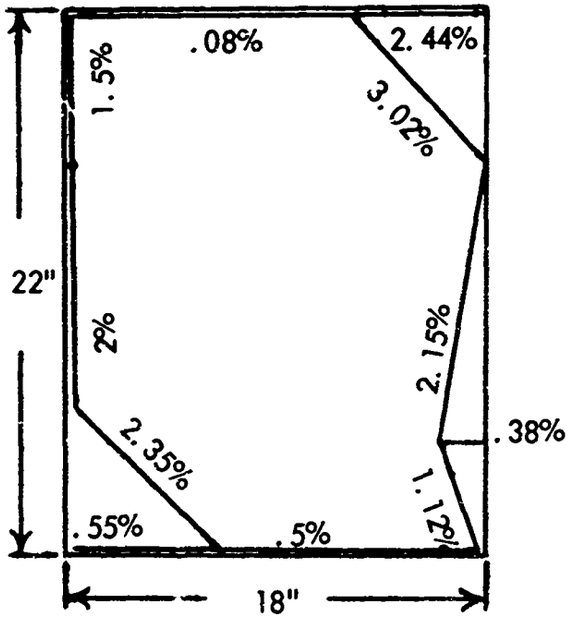
Scale: 1/8" = 1"

FIGURE 10 - A Description of the Flatness Tolerance on Sheet Ta-43-SF-1



Scale: 1/8" = 1"

FIGURE 11 - A Description of the Flatness Tolerance on Sheet Ta-43-SF-2



Scale: 1/8" = 1"

FIGURE 12 - A Description of the Flatness Tolerance on Sheet Ta-43-Uf-1

TABLE 4 - Check Analysis on Wide T-222 Sheets

Element	Analysis (ppm)[1]		
	Ta-43-U. F. -1	Ta-43-S. F. -1	Ta-43-S. F. -2
W	10.6	10.7, 10.6	10.6
Hf	2.58	2.68, 2.66	2.58
C	83	90, 107	90
O	45	44, 45	17
N	10	12, 11	10
Fe	<10	<10, <10	<10
Cr	<10	<10, <10	<10
Ni	<10	<10, <10	<10
Co	<10	<10, <10	<10
Cu	20	(2) 10	10
Mn	<10	<10, <10	<10
Si	<10	<10, <10	<10
Al	<10	<10, <10	<10
Mo	<20	<10, <10	<20
V	<10	<10, <10	<10

(1) Tungsten and hafnium content given in weight percent.

(2) Refer to discussion of chemical analysis (Page 21) for copper result on this sample.

TABLE 5 - Mechanical Property Product Consistency Results on Wide T-222 Sheets.
Material Annealed at 3000°F for 1 Hour.

Heat No.	Room Temp. Transverse Tensile Properties			Bend Properties		
	Ultimate Strength (ksi)	Yield Strength (ksi)	% Elongation (in 1")	1T Weld Bend Transition Temp.	1.6T Weld Bend Transition Temp.	1T Base Metal Bend Transition Temp.
Ta-43-UF-1	123.2	113.5	26			
	121.5	N.D.	24	<-225°F	N.D.	
	121.5	118.5	26	>-250°F	N.D.	<-320°F
Ta-43-SF-1	114.5	113.0	24			
	116.1	113.5	25	<-200°F	N.D.	
	121.5	116.5	27	>-225°F	N.D.	<-320°F
Ta-43-SF-2	121.5	113.5	22			
	123.0	112.5	24	<-200°F	N.D.	
	119.0	111.0	21	>-225°F	N.D.	<-320°F
Ta-37(1,2)	111.1	105.6	25	N.D.	<-250°F >-320°F	N.D.
Ta-39(1,2)	117.2	116.6	23	N.D.	<-320°F	N.D.
Ta-40(1,2)	N.D.	N.D.	N.D.	N.D.	<-250°F >-320°F	N.D.
Ta-43-SF-1(1)	108.7	97.8	27			
	109.2	103.4	30			
	107.1	98.2	30			
	108.3	101.1	23			
	109.4	102.3	24			

(1) Longitudinal tensile results

(2) Average of results entered in Reference 2

N.D. Result not determined

The transverse room temperature tensile data listed in Table 5 for the three Ta-43 sheets show good consistency. The longitudinal tensile data obtained on sheet Ta-43-S. F. -1 is somewhat lower than the corresponding transverse data. The data agree quite closely with results obtained previously on T-222 heat Ta-37. Ductility in both the longitudinal and transverse directions is excellent.

Weld and base metal bend ductility data are also included in Table 5. With a 1t bend radius, the base metal ductile-brittle transition temperature is below -320°F which is typical of T-222. The bend transition temperature of welded samples was about -225°F for heat Ta-43. An exact comparison with previous T-222 data cannot be made because a somewhat larger bend radius was used in the earlier studies. Previous work indicated the weld bend transition temperature range for T-222 to be about -250° to -320°F, which agrees fairly well with the present results when the sharper bend radius is taken into account. The weld bend data clearly indicate that the material produced during the current program exhibits excellent welding characteristics.

Elevated temperature tensile and stress-rupture data obtained on heat Ta-43 from sheet Ta-43-S. F. -1 are summarized in Tables 6 and 7. The results are compared in these tables with data reported previously[2] for heats Ta-37, Ta-39, and Ta-40 produced in earlier programs which contained approximately the same level of solute additions. Comparative chemical analysis data for heats Ta-37, Ta-39, and Ta-40 are shown in Table 8.

The tensile results for heat Ta-43 in the recrystallized condition are internally consistent and reasonably similar to results previously reported on T-222 heats. In general, however, the tensile results on heat Ta-43 are on the low side of the property range established on T-222 produced from smaller heats, corresponding closely with data from heat Ta-37. However, stress-relieved Ta-43 was weaker at 2400°F than material from heat Ta-37 in the equivalent condition. This difference in strength appears to be related to the somewhat lower recrystallization temperature range exhibited by the Ta-43 materials as illustrated in Figure 13. Figure 13 compares the effect of annealing temperature (one hour anneals) on the hardness of as-rolled

TABLE 6 - Comparison* of Elevated Temperature Tensile Results for Several T-222 Heats

Identification	Ultimate Tensile Strength (ksi)	Yield Strength 0.2% Offset (ksi)	% Elongation (in 1 Inch)
Recrystallized 1 Hour at 3000°F - Test Temperature 2000°F			
Ta-37-1	77.6	40.8	18
Ta-37-3	77.8	40.5	20
Ta-39-1	84.5	38.0	19
Ta-40	93.2	49.3	20
Ta-43	81.3	45.0	19
Ta-43	78.0	40.4	18
Test Temperature 2200°F			
Ta-37-1	67.3	38.4	17
Ta-37-3	63.2	36.4	23
Ta-39-1	75.0	39.5	20
Ta-40	82.7	46.2	20
Ta-43	63.7	38.6	24
Ta-43	65.6	39.8	23
Test Temperature 2400°F			
Ta-37-2	53.4	37.8	20
Ta-37-2	51.5	33.4	23
Ta-37-2	48.5	34.8	24
Ta-37-3	48.2	33.3	27
Ta-37-3	46.0	30.7	19
Ta-37-3	47.3	32.3	20
Ta-39-1	59.5	38.2	26
Ta-39-2	58.0	38.0	25
Ta-40	64.0	42.3	23
Ta-40	64.5	38.4	23
Ta-43	51.9	37.4	23
Ta-43	52.5	37.0	25

TABLE 6 - Comparison* of Elevated Temperature Tensile Results for Several T-222 Heats (Continued)

Identification	Ultimate Tensile Strength (ksi)	Yield Strength 0.2% Offset (ksi)	% Elongation (in 1 Inch)
	Test Temperature 2600°F		
Ta-37-1	36.9	29.3	34
Ta-37-3	46.7	34.3	26
Ta-37-3	33.7	29.8	38
Ta-39-1	45.0	34.2	36
Ta-40	50.6	33.5	30
Ta-43	40.0	31.3	26
Ta-43	41.3	32.8	27
	Test Temperature 2800°F		
Ta-37-1	30.5	27.7	48
Ta-37-3	29.5	26.1	34
Ta-39-1	34.0	31.0	68
Ta-40	34.8	33.3	54
Ta-43	31.6	26.6	43
Ta-43	31.2	26.3	47
	Test Temperature 3000°F		
Ta-37-1	24.2	24.0	26
Ta-37-2	24.9	24.1	24
Ta-37-2	24.6	22.8	49
Ta-37-3	15.1	15.1	10
Ta-37-3	17.8	17.6	42
Ta-37-3	23.4	22.4	6
Ta-39-1	26.0	24.6	88
Ta-39-2	28.1	25.6	80
Ta-40	28.2	26.7	73
Ta-40	26.0	25.6	67
Ta-43	23.7	23.5	37
Ta-43	25.2	23.7	32

TABLE 6 - Comparison* of Elevated Temperature Tensile Results for Several T-222 Heats (Continued)

Identification	Ultimate Tensile Strength (ksi)	Yield Strength 0.2% Offset (ksi)	% Elongation (in 1 Inch)
	Test Temperature 3500°F		
Ta-37-1	15.9	15.8	37
Ta-37-2	14.1	14.0	43
Ta-37-3	12.6	11.6	12
Ta-39-1	14.2	--	70
Ta-39-1	13.3	12.8	83
Ta-40	13.7	13.3	48
Ta-43	14.3	14.3	45
Ta-43	15.5	15.5	38
	Stress Relieved 1 Hour at 2000°F - Test Temperature 1800°F		
Ta-37-1	100	89.2	10
Ta-37-3	101	87.2	8
Ta-43	102	90.7	6
Ta-43	102	87.7	6
	Test Temperature 2200°F		
Ta-37-1	78.8	68.8	18
Ta-37-3	91.8	81.8	14
Ta-43	77.7	62.9	21
Ta-43	77.8	69.2	15
	Test Temperature 2400°F		
Ta-37-1	78.8	68.8	18
Ta-37-3	91.8	81.8	14
Ta-43	61.8	47.5	25
Ta-43	58.7	49.5	30

* Results for Heats Ta-37, 39, and 40 obtained from Reference 2. All samples represent the longitudinal test direction.

TABLE 7 - Comparison* of Stress-Rupture Results for Several T-222 Heats

Material Annealed 1 Hour at 3000°F					
Identification	Stress (ksi)	Minimum Creep Rate (%/hr.)	Transition Time (hrs.)	Rupture Time (hrs.)	% Elongation (in 1 Inch)
		Test Temperature 2000°F			
Ta-43	77.5	0.950	1.33	1.75	21
Ta-43	75.0	0.243	2.65	3.80	20
Ta-43	70.0	0.104	7.20	11.00	17
		Test Temperature 2200°F			
Ta-40	65.0	0.420	1.60	3.20	16
Ta-40	60.0	0.180	4.10	8.20	17
Ta-39-1	60.0	0.150	3.90	7.30	19
Ta-43	60.0	1.520	0.57	1.15	21
Ta-43	55.0	1.670	0.35	1.45	21
Ta-39-1	55.0	0.085	4.50	10.00	22
Ta-39-1	55.0	0.094	5.30	11.20	23
Ta-43	50.0	0.279	2.05	5.30	19
Ta-43	45.0	0.810	1.05	8.10	19
		Test Temperature 3000°F			
Ta-37-3	20.0	35.500	0.06	0.26	25
Ta-39-2	17.5	10.600	0.25	1.40	82
Ta-40	17.5	7.400	0.20	1.40	66
Ta-43	17.5	7.900	0.14	1.60	66
Ta-39-2	15.0	4.600	0.30	2.40	94
Ta-40	15.0	3.620	2.10	6.30	68
Ta-43	15.0	2.770	0.15	2.50	75
Ta-37-3	15.0	5.700	0.29	1.10	23
		Test Temperature 2400°F			
Ta-40	45.0	0.87	0.90	3.80	28
Ta-39	42.5	1.07	0.65	3.45	22
Ta-39	40.0	0.56	1.70	7.15	38
Ta-39	40.0	0.61	1.20	6.30	33
Ta-40	40.0	0.29	3.20	10.90	32

TABLE 7 - Comparison* of Stress-Rupture Results for Several T-222 Heats (Continued)

Material Annealed 1 Hour at 3000°F					
Identification	Stress (ksi)	Minimum Creep Rate (%/hr.)	Transition Time (hrs.)	Rupture Time (hrs.)	% Elongation (in 1 Inch)
Ta-37-1	40.0	4.39	1.40	2.15	33
Ta-37-3	40.0	2.55	1.20	2.55	33
Ta-43**	40.0	70.5	0.018	0.075	16
Ta-39	37.5	0.602	2.300	7.000	28
Ta-39	37.5	0.665	1.900	6.90	39
Ta-43	37.5	1.558	1.150	3.25	23
Ta-37-1	35.0	0.520	4.600	12.30	39
Ta-37-3	35.0	0.460	3.200	9.950	39
Ta-43	35.0	0.810	1.050	3.900	18
Ta-37-3	32.5	0.129	5.600	15.600	31
Ta-37-3	32.5	0.048	8.600	26.000	31
Ta-43	32.5	0.247	2.900	11.85	35
Ta-43	30.0	0.088	5.10	26.20	28
Ta-43	30.0	0.126	6.40	21.30	27

* Results for heats Ta-37, 39, and 40 obtained from Reference 2.

** Sample used for a second check on chemical analysis of the sheet.

TABLE 8 - Chemical Analysis for T-222 Comparison Heats

Heat No.	Sample	Analysis (w/o)		Analysis (ppm)											
		W	Hf	C	O	N	Cb	V	Si	Cr	Ni	Ti	Cu	Al	Mg
Ta-37	Ingot	9.5	2.4	110	23	12	--	--	--	--	--	--	--	--	--
	Sheet	9.6	2.4	120	29	25	2300	100	5	20	30	5	1	40	2
Ta-39	Ingot	8.9	2.2	130	18	41	--	--	--	--	--	--	--	--	--
	Sheet	9.2	2.2	130	--	--	100	10	10	20	30	5	1	40	2
Ta-40	Ingot*	11.0	2.5	120	34	--	--	--	--	--	--	--	--	--	--

* Chemistry on sheet from this heat was not obtained.

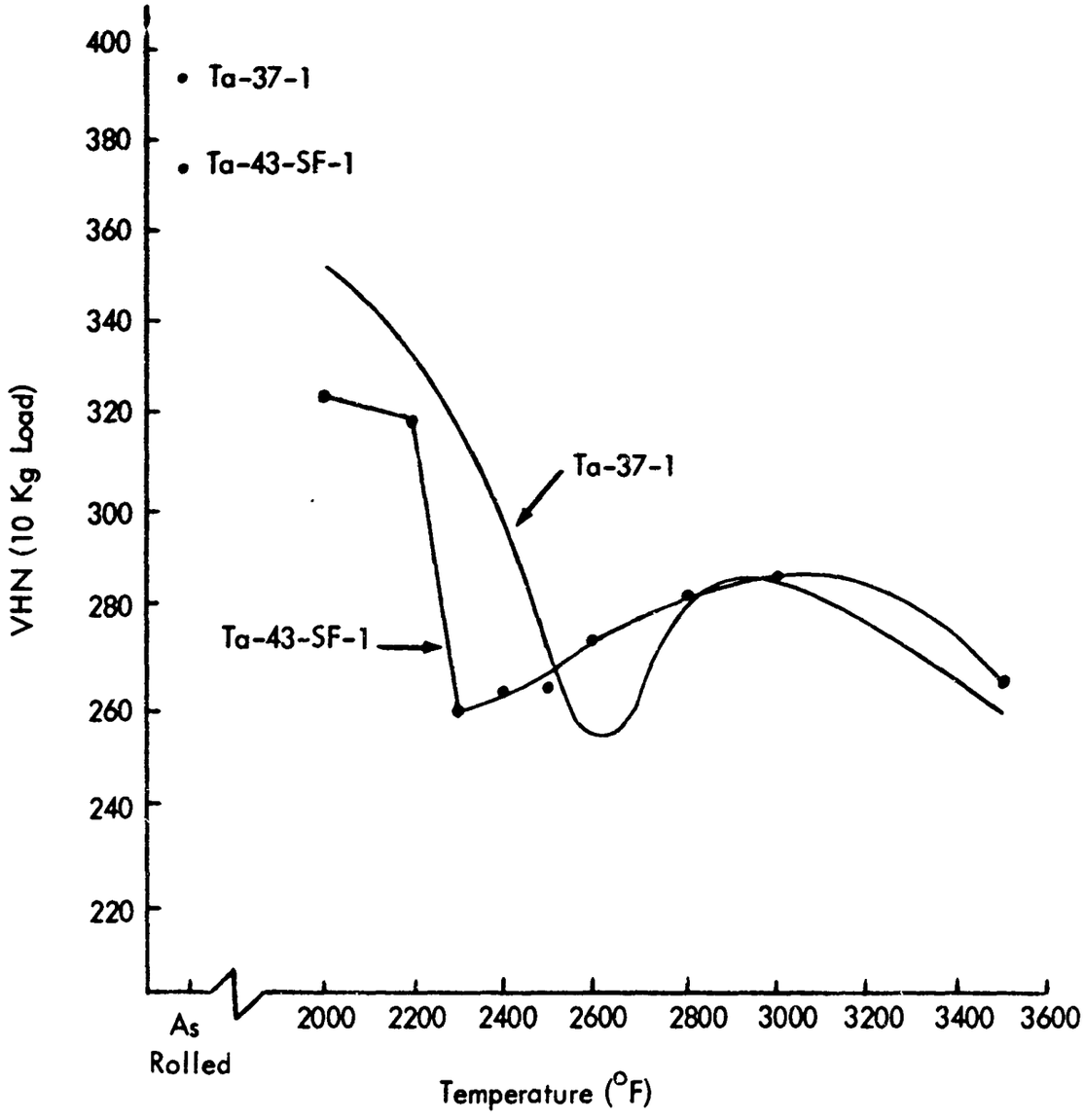
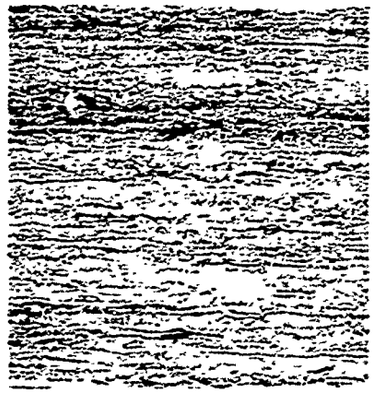


FIGURE 13 - A Comparison of Room Temperature Hardness as a Function of One Hour Annealing Temperature for Heats Ta-37 and Ta-43

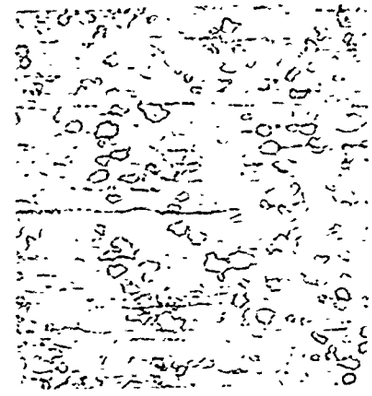
material from heats Ta-37 and Ta-43. The data indicate a lower recrystallization temperature for Ta-43. This is confirmed by the results of metallographic and x-ray examination shown in Figures 14 and 15. Metallographic examination indicated recrystallization beginning at 2300°F for Ta-43, while the Laue back reflection patterns of Figure 15 show that recrystallization is complete after a 1 hour anneal at 2600°F, as opposed to 2800°F for Ta-37.[2] These data indicate that recrystallization was more extensive during testing stress-relieved Ta-43 at 2400°F, as compared to Ta-37 in the same condition, resulting in lower strength and higher elongation for Ta-43.

Stress-rupture data are listed in Table 7 and summarized in Figure 16. The results of the first sample to be tested, at 40,000 psi and 2400°F, deviated significantly from the data on previous heats. Since the stress-rupture bars were taken from sheet Ta-43-S. F. -1 which displayed a high copper content (60 ppm) this element was immediately suspected as the cause for premature failure, and this stress-rupture bar was used for a second check analysis sample. As described previously, neither copper nor any other potentially detrimental element was found to be in excess of its expected level on this sample. An explanation for the anomalous behavior of the sample was not obtained. However, this was the only specimen to show properties markedly different than normal.

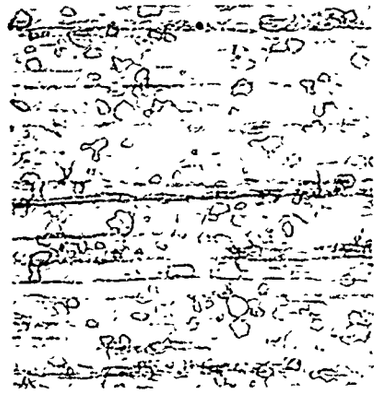
All other results on Ta-43 are seen to be consistent within the heat, and show the expected temperature-stress trend. Heat Ta-43 does, however, display somewhat shorter rupture life, and increased secondary creep rates as compared to previous heats. The greatest amount of stress-rupture data for T-222 was obtained at 2400°F, allowing for best comparison of properties to be made at this temperature. Again, as observed in the tensile data comparisons, heat Ta-43 corresponds closest to Ta-37. It is unlikely that the poorer stress-rupture properties of heat Ta-43 are due to some unusual containment which was not analyzed for, but instead these results are probably more indicative of the scatter one obtains when comparing heats of varying ingot size etc. For example, stress-rupture data for heat resistant alloys are often characterized by scatter bands 10 ksi in width.[4]



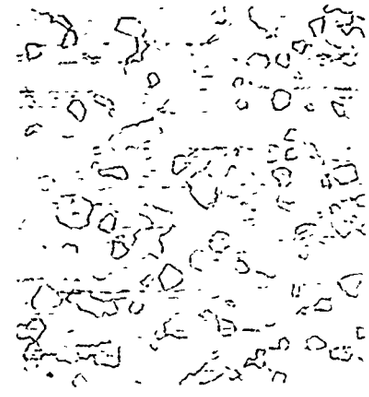
1 hr at 2200°F



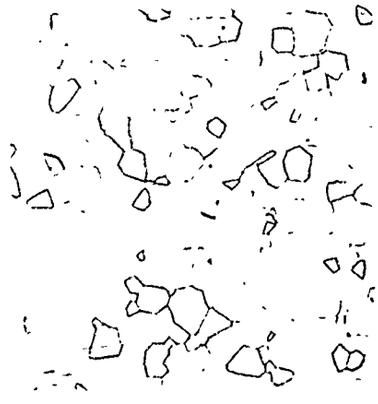
1 hr at 2300°F



1 hr at 2600°F



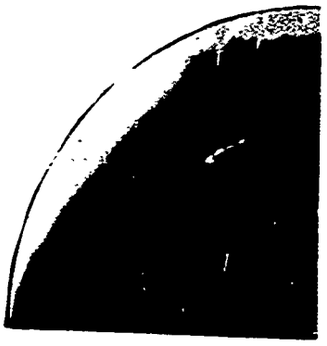
1 hr at 2800°F



1 hr at 3000°F

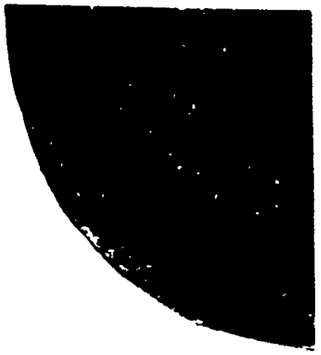
1 hr at 3500°F

FIGURE 14 - Microstructure of Sheet Ta-43-SF-1 After Various Heat Treatments (100X)



1 Hr. at 2400°F

1 Hr. at 2500°F



1 Hr. at 2600°F

1 Hr. at 2800°F

FIGURE 15 - Laue X-Ray Patterns of Sheet Ta-43-SF-1
After Various Heat Treatments

The one and ten hour rupture stress for these heats are given in Table 9. Also included in the table for comparative purposes is the rupture data for T-111 alloy.[1] A Larson-Miller plot for all T-222 heat stress-rupture data is given in Figure 17. The data band shown in this figure is the best description of the stress-rupture properties of T-222 available to date.

The property comparisons described above show that the scale-up to 4 inch ingot and subsequent production of wide sheet did not significantly alter the strength, ductility, and weldability of T-222. In addition, the excellent fabricability of T-222 was retained in scale-up as evidenced in the high quality sheet bar obtained by Dynapak forging as-cast ingot, and the good ingot-to-sheet yield of approximately 35%.

V. PHASE IDENTIFICATION AND METALLOGRAPHY

Even though the carbon content in T-222 alloy is relatively low (~100 ppm), the alloy is responsive to heat treatment in the sense that it can be solution treated to yield a single phase microstructure, or it can be aged to precipitate second phases. As a result of the observed strengthening effects due to interstitial additions of C and N to the base T-111 alloy,[1] and because of the possibility of exploiting precipitation hardening effects, a study was initiated to identify the second phases present in T-222, for various mechanical-thermal conditions. Two main mechanical-thermal treatments were studied: In one case the material was solution treated at 3000-4000°F, quenched, and aged; in the other main treatment the as-rolled material was aged directly.

Although the alloys studied are quaternary Ta-W-Hf-C alloys, the results are conveniently rationalized in terms of the ternary Ta-Hf-C system, i. e., it is assumed that W merely substitutes for Ta and Hf in the phases occurring in the ternary system. Rudy[5] has recently published the complete phase diagram for the Ta-Hf-C system and an isothermal section of this diagram is shown in Figure 18. The phases which are observed in the Ta-corner of the diagram are: (1) the BCC (Ta,Hf,C) solid solution, designated M, (2) the HCP dimetal carbide $(Ta,Hf)_2C$, designated M_2C and which contains about 10 at % Hf, and (3) the FCC monometal

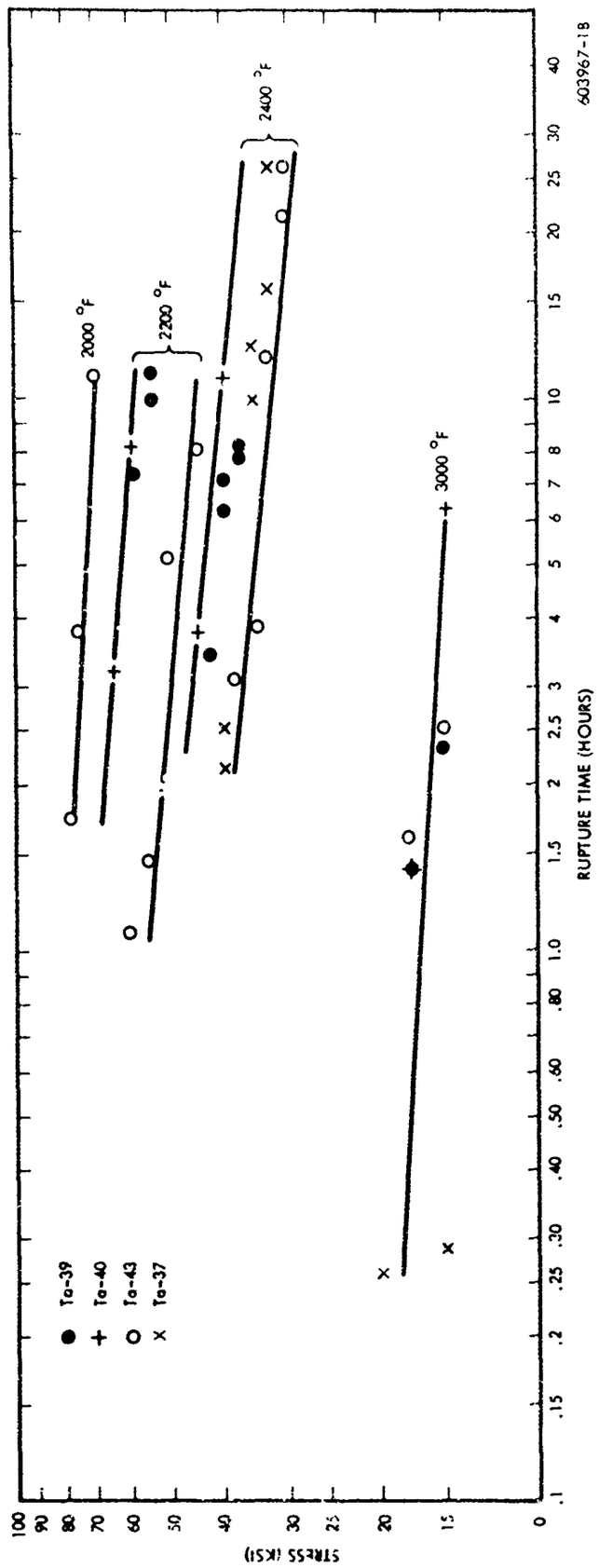


FIGURE 16 - Stress-Rupture Properties of T-222 Heats Ta-37, Ta-39, Ta-40, and Ta-43. Material Annealed 1 Hour at 3000°F.

TABLE 9 - One and Ten Hour Stress-Rupture Properties for Various T-222 and T-111 Heats

Alloy	Heat No.	Temperature (°F)	Stress for 1 Hr. Life (ksi)	Stress for 10 Hr. Life (ksi)
T-222	Ta-43	2000	80	70
T-222	Ta-43	2200	56	45
T-222	Ta-39	2200	65	55
T-222	Ta-40	2200	72	69
T-222	Ta-37	2400	43	35
T-222	Ta-39	2400	47	39
T-222	Ta-40	2400	52	40
T-222	Ta-43	2400	42	32
T-111	---	2400	33.5	26
T-222	Ta-37	3000	15	9
T-222	Ta-39	3000	18	12
T-222	Ta-40	3000	18	14
T-222	Ta-43	3000	18	12

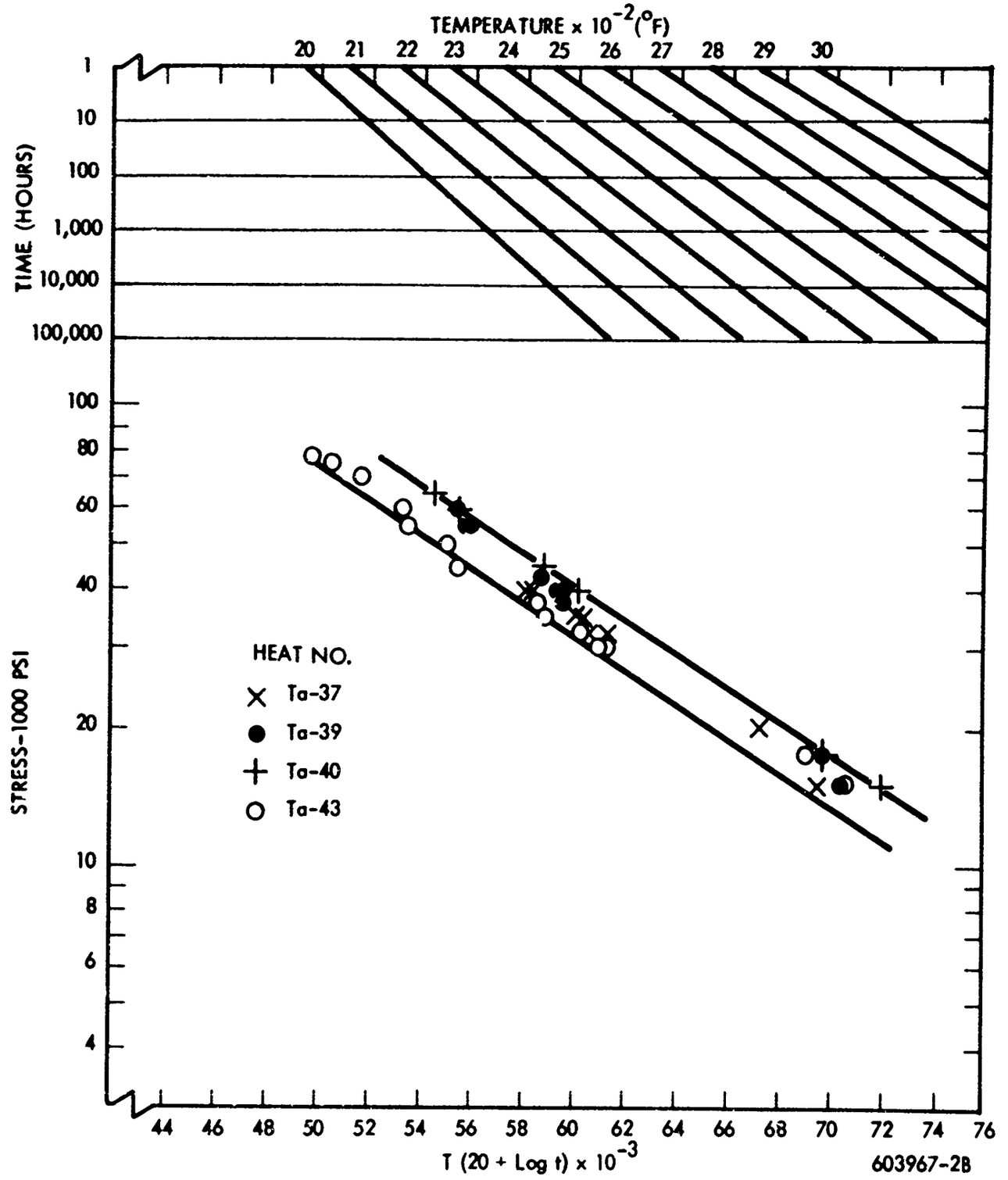


FIGURE 17 - Larson-Miller Plot of Stress-Rupture Data for Various T-222 Heats. Material Annealed 1 Hour at 3000°F

carbide (Ta,Hf)C, designated MC and which represents the series of solid solutions formed between TaC and HfC. These are the three phases, neglecting HfO_2, observed in the experimental alloys, but with some W included in each phase. The point P in the Ta-corner of the diagram of Figure 18 moves towards the Ta-corner with decreasing temperature and at 2000°F falls at about 2 at % Hf.

The results obtained on two heats of T-222 (~100 ppm C, heats Ta-43 and Ta-39) and a heat modified to contain 400 ppm C (heat Ta-42) are shown in Table 10. Similar results were obtained on other heats of T-222 as well as a heat of T-111 modified to contain 100 ppm of each C, O, and N. The experimental results are shown graphically in Figure 19. This figure shows a schematic drawing of the Ta-corner of the Ta-Hf-C system, upon which the experimental data are superimposed. There are two sets of points plotted for each of the alloys; one set shows the results obtained after aging the as-rolled material directly; the other set displays the results obtained after aging solution treated material. Heats representing T-222 chemistry i.e., Ta-39 and Ta-43 will be discussed first.

With the processing schedule used, heat Ta-39, was single phase in the as-rolled condition, and aging the as-rolled material for 16 hours at temperatures between 2000 and 2800°F caused precipitation of only the MC phase. Annealing Ta-39 at higher temperatures, 1 hour at 3000 to 4000°F, gave a single phase structure which could be retained by He-quenching, i.e., quenching in a stream of He gas. The response to aging as-rolled material from Ta-43 was examined only at 2000°F whereupon monocarbide precipitation was also detected.

Aging the solution treated material (quenched from 3000 to 4000°F to give a single phase structure) at 1600 to 2000°F caused precipitation of only the M_2C phase. Aging at 2400°F precipitated either both the M_2C and MC phases (Ta-39) or just the MC phase (Ta-43) which undoubtedly is a result of the slightly differing carbon contents. After aging at 2800°F the material was either single phase (Ta-43) or showed precipitation of the MC phase (Ta-39), and again this is probably related to the exact carbon content of the material.

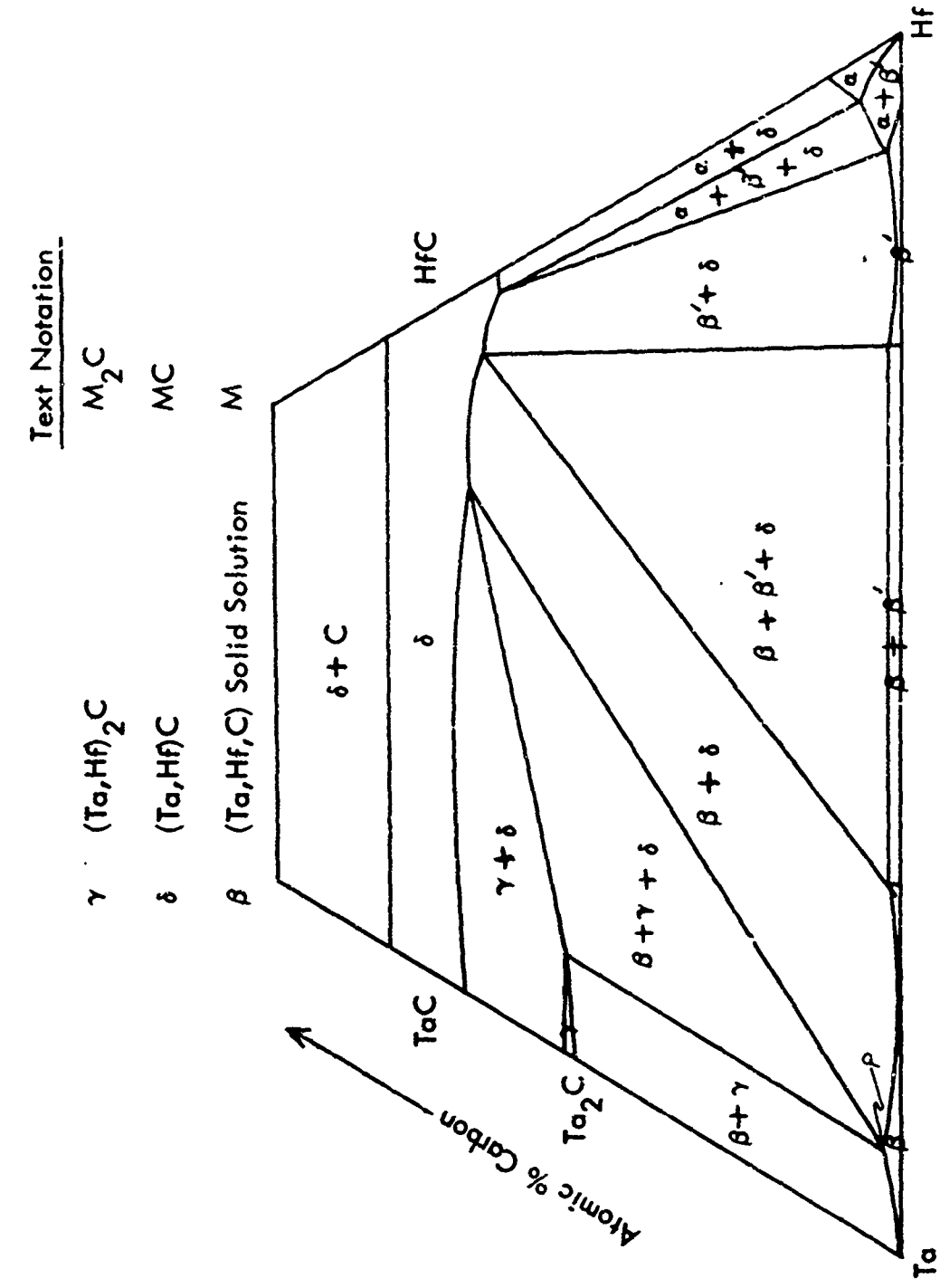


FIGURE 18 - An Isothermal Section Through the Ta-Hf-C System at 3182°F as Reported by Rudy *et al*[5]

Since the precipitates observed after aging at the lower temperatures (1600 to 2000°F) were different for the as-rolled material (MC) and the solution treated material (M_2C) a few additional tests were run to clarify this influence of mechanical-thermal history. These tests were run on heat Ta-43 and the results are also shown in Table 10. Sample X25 was solution treated, strained 5% at room temperature, and then aged at 2000°F. Only the MC phase precipitated, which shows that a small amount of strain induces precipitation of the MC phase at the lower temperature where only the M_2C phase precipitates in the absence of strain. This effect is reversible, as shown by the results on Sample X31. The as-rolled sample was aged at 2000°F to precipitate the MC phase, then solution treated and reaged at 2000°F. The MC phase formed during the first aging treatment dissolved during the solution treatment, the prior strain was removed by recrystallization, and only the M_2C phase precipitated during the final aging treatment. Sample T7 was run with the aim of possibly determining the equilibrium phases at 2000°F. The sample was solution treated and aged at 2400°F to precipitate the MC phase, quenched and reaged at 2000°F. Both the MC and M_2C phases were observed after the 2000°F aging treatment, but the MC phase was much more abundant as evidenced by the relative intensities of the diffraction patterns. The presence of the M_2C phase suggests the possibility that the MC phase was dissolving while the M_2C phase was precipitating as the equilibrium phase, but that the reaction did not go to completion because of slow kinetics. Suffice it here to say that this interpretation meets with several objections, and so the equilibrium phases at 2000°F remain unknown.

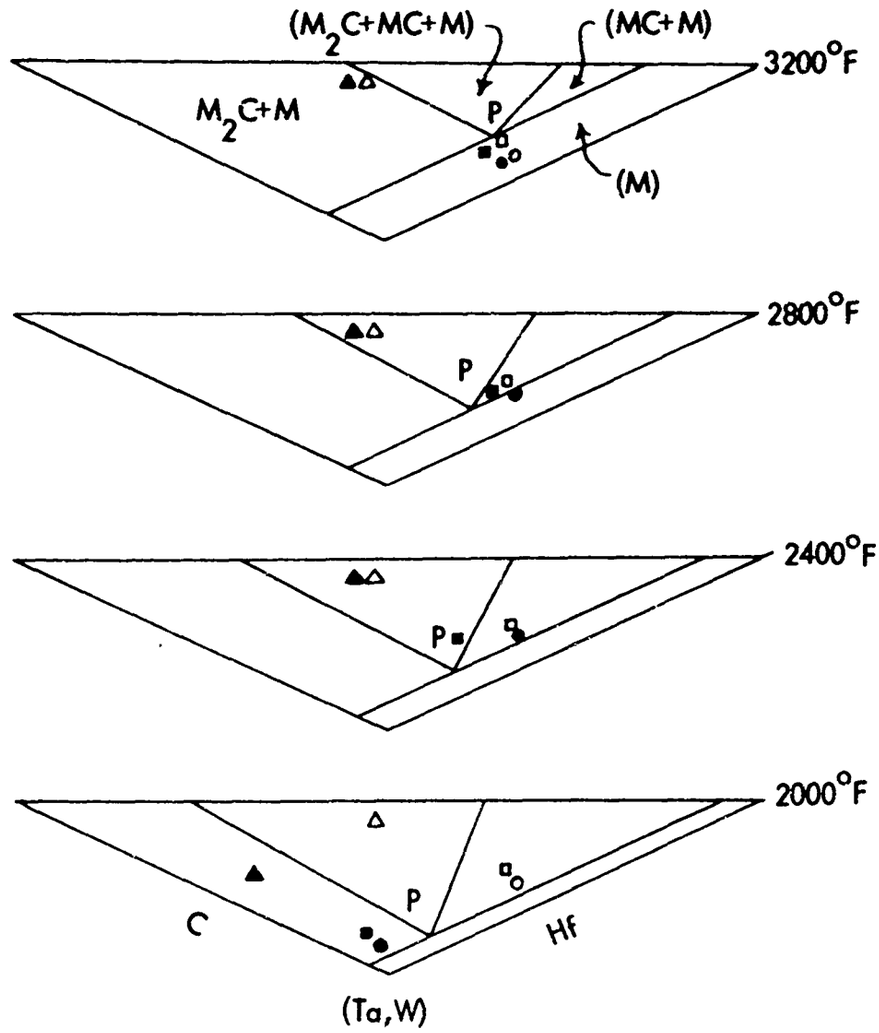
Referring to the 400 ppm C heat, (Ta-42), the as-rolled material contained both the M_2C and the MC second phases. Both second phases were still observed after aging the as-rolled material at 2000 to 2800°F, although the relative amount of the MC phase appeared to increase during aging. The MC phase dissolved upon annealing for 1 hour at 3000 to 4000°F, but the M_2C phase remained. After quenching from the high temperature and aging at 2000°F the M_2C phase was still the only second phase observed, and presumably additional precipitation of M_2C occurred during aging. Aging the quenched material at 2400 to 2800°F caused precipitation of the MC phase since both second phases were observed after these aging treatments.

TABLE 10 - Carbide Phases Detected in Ta-W-Hf-C Alloys

Mechanical-Thermal Condition	Heat Ta-39 Ta-9.2W-2.2Hf -.013C	Heat Ta-43 Ta-10.7W-2.7Hf -.010C	Heat Ta-42 Ta-9.4W-2.4Hf -.040C
Sol. Treated 1 or 16 hr/3000-4000°F + Quench*	N. D.	N. D.	M_2C
As-Rolled (500°F)	N. D.	--	$M_2C^{(A)}+MC^{(L)}$
Sol. Treated 1 hr/3000°F + Aged 16 hrs. at: 1600°F	--	M_2C	--
2000°F	M_2C	M_2C	M_2C
2400°F	$M_2C^{(M)}+MC^{(A)}$	MC	$M_2C^{(A)}+MC^{(L)}$
2800°F	MC	N. D.	$M_2C^{(A)}+MC^{(M)}$
As-Rolled (500°F) + Aged 16 hrs. at: 2000°F	MC	MC	$M_2^{(M)}+MC^{(A)}$
2400°F	MC	--	$M_2C^{(M)}+MC^{(A)}$
2800°F	MC	--	$M_2C^{(M)}+MC^{(A)}$
Sample X-25			
1 hr/3000°F+Roll (5% at 500°F) + Aged 16 hrs/2000°F	--	MC	--
Sample X-31			
Roll (90% at 500°F) + 16 hrs/2000°F + 1 hr/3000°F + 16 hrs/2000°F	--	M_2C	--
Sample T-7			
1 hr/3000°F + 16 hrs/2400°F + 16 hrs/2000°F	--	$M_2C^{(MW)}+MC^{(S)}$	--

* Specimens were quenched in a helium stream between all heat treatments.

MC: (HF,Ta,W)C, M_2C: $(Ta,W,Hf)_2C$, N. D.: not detected, (S) strong, (A) abundant, (L) low, (M) moderate, (MW) moderate to weak, (--) not analyzed.



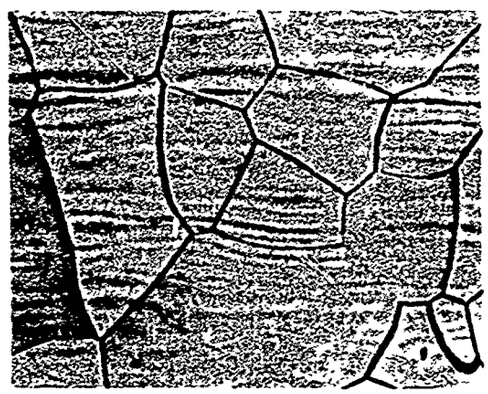
Key	Heat No.	Carbon Analysis (ppm)	Thermal-Mechanical Treatment Prior to Aging
□	Ta-39	130	Cold Rolled (5000°F) + 16 Hr. Age
△	Ta-42	400	
○	Ta-43	100	
■	Ta-39		Cold Rolled (500°F) + Solution Treat 1 Hr./3000°F (Structure Equivalent to that Shown at 3200°F) + 16 Hr. Age
▲	Ta-42		
●	Ta-43		

FIGURE 19 - Phases Present in Ta-W-Hf-C alloys as a Function of Thermal-Mechanical History

X-ray emission analyses on the extracted residues showed that the MC phase was rich in Hf and Ta (much richer in Hf than Ta) but low in W, whereas the M_2C phase was rich in Ta and W (richer in Ta than W) but low in Hf. The lattice parameter of the FCC MC phase was approximately $a_o = 4.60$, which, in agreement with the x-ray emission analyses, corresponds to about 75% HfC, assuming a linear variation in lattice parameter between TaC and HfC. The lattice parameters for the HCP M_2C phase were approximately $a_o = 3.10$, $c_o = 4.94$, and $c/a = 1.59$, which are close to the values reported for Ta_2C. Chemical analyses were not made to determine interstitial contents, and it is possible that the cubic phase contains N, and perhaps O, as well as C. However, as shown in Table 10, the compositions of the second phases are listed as (Hf,Ta,W)C for the FCC phase, otherwise designated as MC, and as $(Ta,W,Hf)_2C$ for the HCP phase which is designated as M_2C.

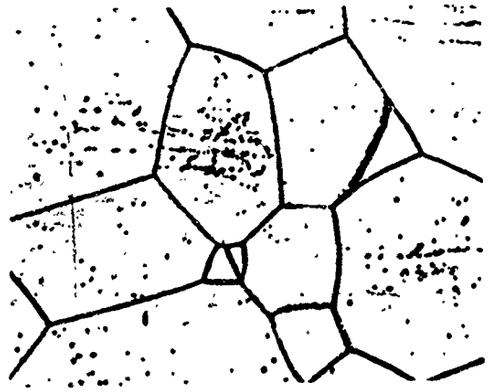
Repeated tests have shown that the phases observed after aging at 2000°F or lower depends upon the mechanical-thermal history immediately prior to aging, but with this exception the ternary Ta-Hf-C phase diagram provides a ready physical basis for correlating the experimental results. Several explanations might be advanced to explain the low temperature results. Assuming that the MC carbide is stable at 2000°F, the presence of the M_2C phase might simply be due to its preferential nucleation at grain boundaries, as evidenced by metallographic observations. Thus, once formed the M_2C phase might persist in spite of greater assumed stability of the MC phase. On the otherhand, if the M_2C phase is the only stable second phase at 2000°F, the presence of the cubic MC phase might be due to its stabilization by residual oxygen and nitrogen, as proposed by Chang[6] who investigated alloys similar to T-222.

The microstructures and precipitate morphologies in T-222 alloy, (heat Ta-43), after various mechanical-thermal treatments are shown in Figures 20 and 21. The material is single phase in the solution treated condition, Figure 20A. The HCP M_2C phase formed by aging the solution treated material at 1600-2000°F precipitates preferentially at grain boundaries, Figure 20B, and sometimes as a continuous, massive phase up to 3-4μ wide, Figure 21A. Even as matrix precipitates, the M_2C particles are generally large, and appear to be platelet in shape, Figure 21B. The FCC MC precipitates formed by aging the solution treated material



(a)

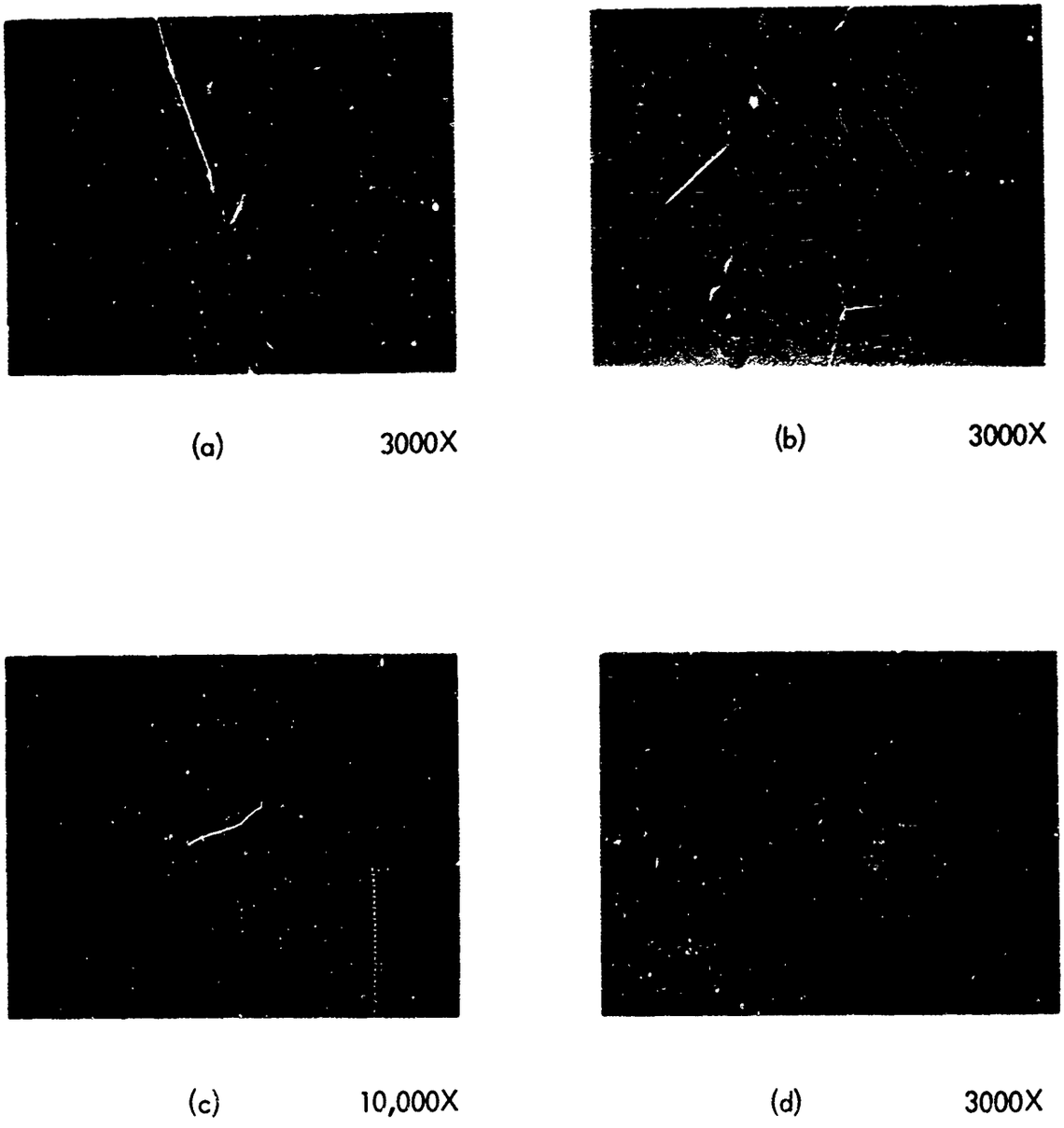
(b)



(c)

(d)

FIGURE 20 - Microstructures of T-222 Alloy. (a) Solution Treated 1 Hr/3000°F, He-Quenched (Single Phase). (b) Solution Treated + Aged 16 Hrs/1600°F (M_2C ppt). (c) Solution Treated + Aged 16 Hrs/2400°F (MC ppt). (d) As-rolled + Aged 16 Hrs/2000°F (MC ppt). (400X Mag.)



(a) 3000X (b) 3000X

(c) 10,000X (d) 3000X

FIGURE 21 - Electron Micrographs of Surface Replicas of T-222 Alloy. Solution Treated + Aged 16 Hrs. at: (a) 2000°F, M_2C ppt, (b) 1600°F, M_2C ppt, (c) and (d) 2400°F MC ppt.

at 2400°F and the as-rolled material at 2000°F are shown in Figures 20C and D, respectively. In the wrought and aged material, the precipitates are located primarily in high angle boundaries. In the solution treated and aged material, the MC precipitates form throughout the matrix and along grain boundaries. The grain boundary precipitates occur as discrete and somewhat uniformly spaced particles, but there is no general preference for grain boundary precipitation. The MC precipitates are small, less than 1μ in size, and are heterogeneously distributed throughout the matrix. At low magnification these precipitates appear round in shape, but surface replicas and transmission micrographs of extracted particles show that they are platelets. Figures 21C and D show MC platelets in the matrix and along subgrain boundaries, respectively.

VI. MICROSTRUCTURE AND MECHANICAL PROPERTIES

In the previous section of this report it was shown that T-222 is responsive to heat treatment, in that the phases observed in the microstructure are influenced by mechanical-thermal history. To evaluate the effect of prior mechanical-thermal treatment on the properties of T-222, tensile tests were conducted on material from heats Ta-39 and Ta-43 in various conditions of heat treatment. These tests were intended to determine the following:

1. The effect of solution annealing temperature on room temperature tensile properties.
2. The effect of aging temperature on the room temperature tensile properties of solution annealed sheet.
3. The effect of prestraining plus aging on room temperature tensile properties.
4. The effect of intermediate annealing on 2400°F tensile properties.

EFFECT OF SOLUTION ANNEALING AND AGING

Room and 2000°F tensile data for heat Ta-43 in various conditions of heat treatment are summarized in Table 11. The effect of solution annealing temperature on room temperature

tensile properties is shown in Figure 22 (specimens 1 thru 10, Table 11). Strength decreased as the solution annealing temperature was increased from 3000 to 4000°F. Tensile elongation decreased rather abruptly as the annealing temperature was raised from 3500 to 4000°F. This variation in strength and ductility is probably related to the variation in grain size with solution annealing temperature. Referring to Table 11, specimens 6, 7, and 8 were used for pre-straining and aging experiments, hence only the yield strength was obtained for the solution annealed condition. Specimen 10 was radiation cooled in the furnace, whereas the other specimens were all quenched in a stream of helium gas. No variation in strength or ductility was observed as a result of this variation in cooling rate.

Room temperature tensile results on material solution annealed and aged are shown in Figure 23. Heat Ta-43, was single phase after aging at 2800°F, hence the tensile properties of the specimens aged at this temperature are no different from those of the as-solution annealed material. However, aging for 1 and 16 hours at temperatures ranging from 1600 to 2400°F resulted in a decrease in room temperature strength (specimens 11 thru 18). Aging solution treated material at 2000°F also caused a decrease in 2000°F tensile strength (compare specimens 3A, 12A and 16A, Table 11). It is thus apparent that precipitation of both the MC and M_2C carbide phases, which resulted from the aging treatments, caused a decrease in tensile strength. As shown in Figure 23 aging for 16 hours caused a somewhat greater reduction in strength at any given temperature than the corresponding 1 hour aging treatment.

Aging as-rolled material at 2000°F caused a decrease in room temperature strength, but had no effect on 2000°F tensile strength (specimens 19, 19A, 20, and 20A).

Specimens 21 thru 24 were processed to explore the possibility of achieving strain-induced precipitation. Specimen 21 was solution treated and then creep strained for 24.4 hours at 2000°F and 42 ksi to a plastic strain of 1%. Following this treatment, the specimen was tested in tension at room temperature. The room temperature strength of this specimen was about 10 ksi lower than that of the solution treated material: however, it was also about 10 ksi higher than that of the specimen (16) aged for 16 hours at 2000°F. Specimens 22, 23, and 24 were solution treated at 3000°F, pre-strained at room temperature (see the yield

TABLE 11 - Room Temperature Tensile Properties of Heat Treated T-222 (Heat Ta-43)

Mechanical-Thermal Treatment	Specimen No. ***	Room Temp. Tensile Results			2000°F Tensile Results		
		UTS (ksi)	.2% YS (ksi)	Elong. (%)	UTS (ksi)	.2% YS (ksi)	Elong. (%)
ST 1 Hr./4000°F	1	89.4	89.4	4.0			
ST 1 Hr./3500°F	2	100.4	94.6	32.0			
ST 1 Hr./3000°F	3-3A	108.7	97.8	27.0	78.2	39.0	20
ST 1 Hr./3000°F	4	109.2	103.4	30.0			
ST 1 Hr./3000°F	5	107.1	98.2	30.0			
ST 1 Hr./3000°F	6	--	101.0	--			
ST 1 Hr./3000°F	7	--	99.1	--			
ST 1 Hr./3000°F	8	--	113.1	--			
ST 1 Hr./3000°F	9	108.3	101.1	23.0			
ST 1 Hr./3000°F	10**	109.4	102.3	24.0			
ST* + Aged 1 Hr./2800°F	11	107.6	99.2	23.0			
ST + Aged 16 Hrs./2800°F	12-12A	104.9	97.2	31.0	78.2	39.6	23
ST + Aged 1 Hr./2400°F	13	102.2	92.1	29.0			
ST + Aged 16 Hrs./2400°F	14	101.4	88.4	30.0			
ST + Aged 1 Hr./2000°F	15	106.0	92.8	25.0			
ST + Aged 16 Hrs./2000°F	16-16A	97.7	81.7	24.0	65.8	34.5	18
ST + Aged 1 Hr./1600°F	17	102.4	87.6	25.0			
ST + Aged 16 Hrs./1600°F	18	100.8	85.7	28.0			
As-Rolled	19-19A	168.2	156.1	6.0	96.0	73.4	14
As-Rolled + Aged 16 Hrs./2000°F	20-20A	131.9	123.4	17.0	90.0	73.8	13
ST + Creep strain 1% at 2000°F (24.4 Hrs., 42 ksi)	21	99.9	90.8	27.0			
ST + strain 6% Room Temperature 16 Hrs./2000°F	22	105.0	94.5	29.0			
ST + strain 10% Room Temperature 16 Hrs./2000°F	23	108.9	98.3	31.0			
ST + strain 7% Room Temperature 16 Hrs./2000°F	24	119.6	108.4	31.0			

* ST = Solution Treated 1 Hr./3000°F + He-Q

** All specimens were He-Q after annealing or aging except S11 which was radiation cooled in the furnace.

*** Intergers refer to room temperature tests. Intergers plus letter refer to test at 2000°F.

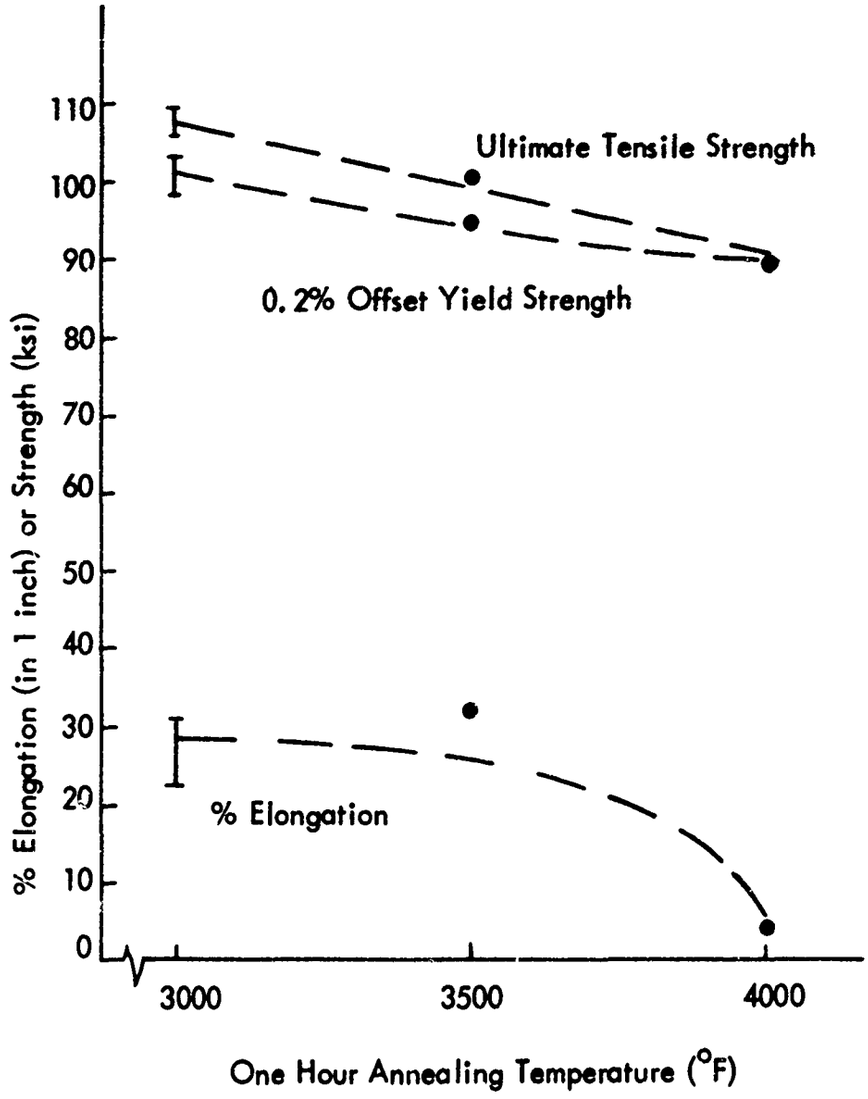


FIGURE 22 - Effect of Solution Annealing Temperature on Room Temperature Mechanical Properties of T-222 (Heat Ta-43)

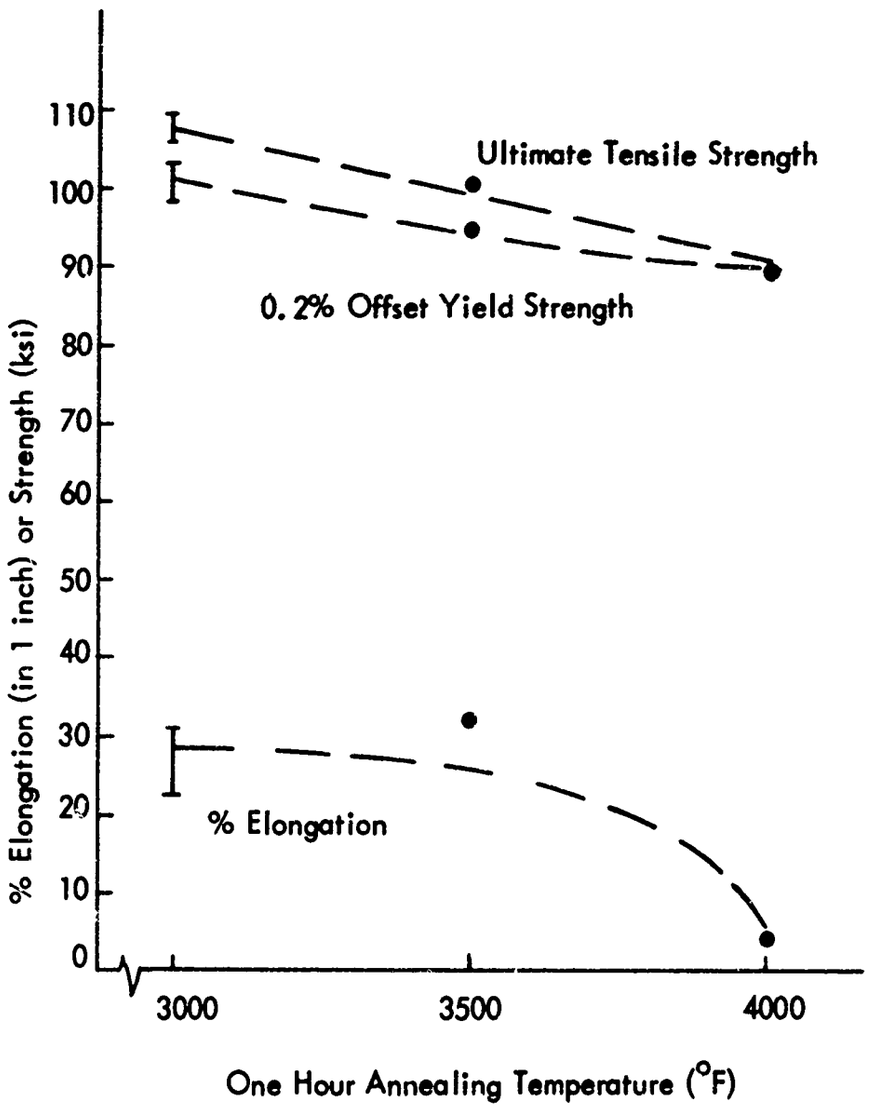


FIGURE 22 - Effect of Solution Annealing Temperature on Room Temperature Mechanical Properties of T-222 (Heat Ta-43)

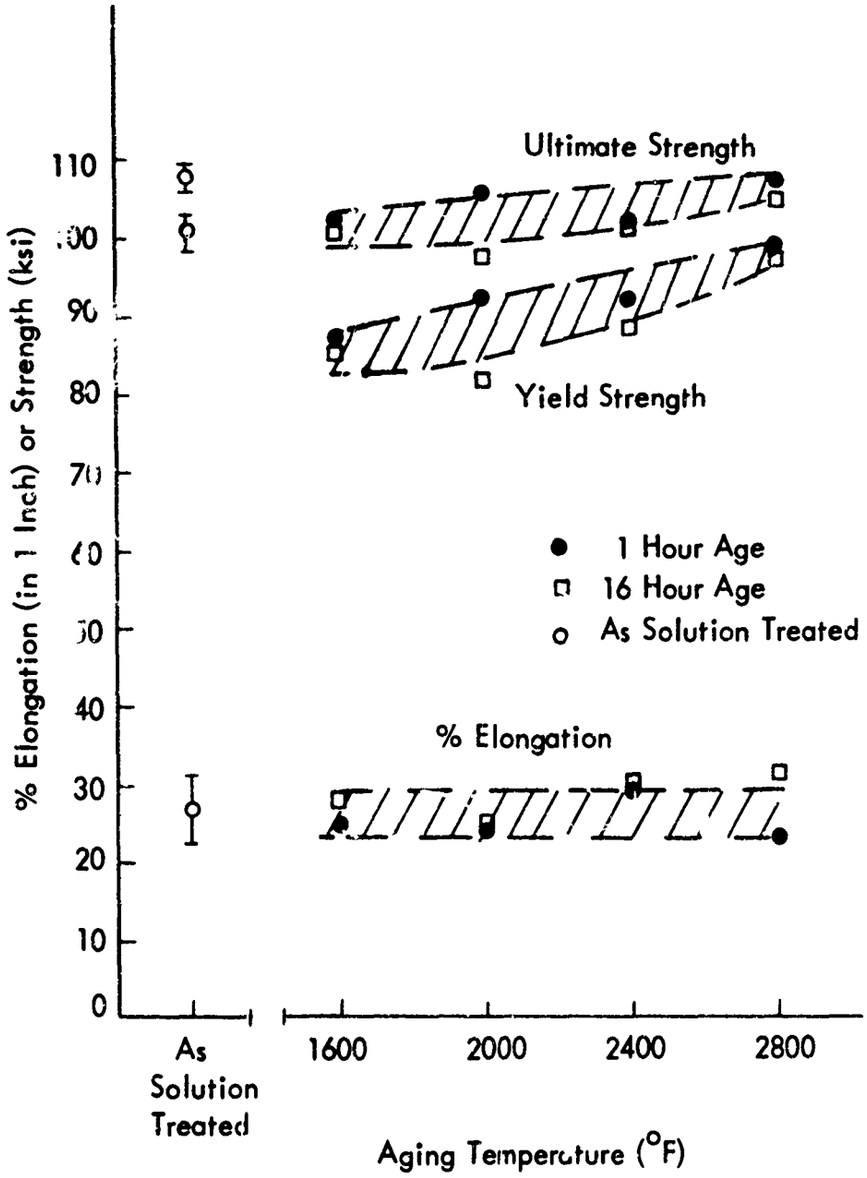


FIGURE 23 - Effect on Room Temperature Mechanical Properties of T-222. Material Solution Treated Prior to Aging for 1 Hr. at 3000°F (Heat Ta-43)

strengths for specimens 6, 7, and 8, respectively) aged 16 hours/2000°F, and then retested at room temperature. The room temperature yield strength of the specimens pre-strained 6 and 7% and then aged (22 and 24) was about 5 ksi lower than the yield strength of the same specimens prior to pre-straining and aging (6 and 8). The strength of specimen 8, or equivalently specimen 24, was about 10 ksi higher than that of other similarly heat treated specimens, but the reason for this is not known. The strength of specimen 23 after pre-straining about 10% and aging was about the same as that observed prior to pre-straining, specimen 7.

For the thermal-mechanical conditions studied, the tensile test results show that in the recrystallized condition T-222 is strongest when solution treated to give a single phase microstructure as judged by metallographic examination. Aging the solution treated material at temperatures between 1600 and 2400°F to give the type of microstructures (precipitate morphology) shown in Figures 20 and 21 results in a decrease in room temperature strength, and based upon specimen 16A, strength at 2000°F also. The decrease in strength due to aging was not as great if the specimen was pre-strained prior to aging. Pre-straining a sufficient amount, perhaps about 10%, prior to aging resulted in the same strength as that of the solution treated material. In no cases, however, did aging or pre-straining plus aging result in increased strength above that of the initially solution treated material.

EFFECT OF INTERMEDIATE THERMAL-MECHANICAL TREATMENTS

To explore the effect of intermediate processing treatments on the high temperature strength of T-222, a series of tensile specimens were prepared from heat Ta-39 of varying thermal-mechanical history. The processing of this material is illustrated in Figure 24. (Melting data for this heat is presented in Reference 2). After forging and working this heat to 0.1 inch thick plate, five 1-3/4 inch by 6 inch strips were removed and fabricated to 0.045 inch thick sheet using the procedures listed below:

A. Cold rolled directly to 0.045 inch thick (~80% red.).
B. Annealed 1 hour at 3000°F, cold rolled to 0.045 inch thick (55% red.).
C. Annealed 1 hour at 3500°F, cold rolled to 0.045 inch thick (55% red.).

D_1 Annealed 1 hour at 3500°F, cold rolled to 0.06 inch (40% red.), annealed 1 hour at 3000°F, cold rolled to 0.045 inch (25% red.).

D_2 Annealed 1 hour at 3500°F, cold rolled to 0.060 inch (40% red.), annealed 1 hour at 3500°F, cold rolled to 0.045 inch (25% red.).

Each strip was then sheared into four sections and annealed 1 hour at 2600, 2800, 3000, and 3500°F, with the exception of D_1 and D_2 which were not annealed at 2600°F. The 2600°F anneal was omitted because of the low reduction of 25% which would not be sufficient to cause recrystallization at that temperature. In this manner a number of thermal-mechanical treatments prior to aging were obtained.

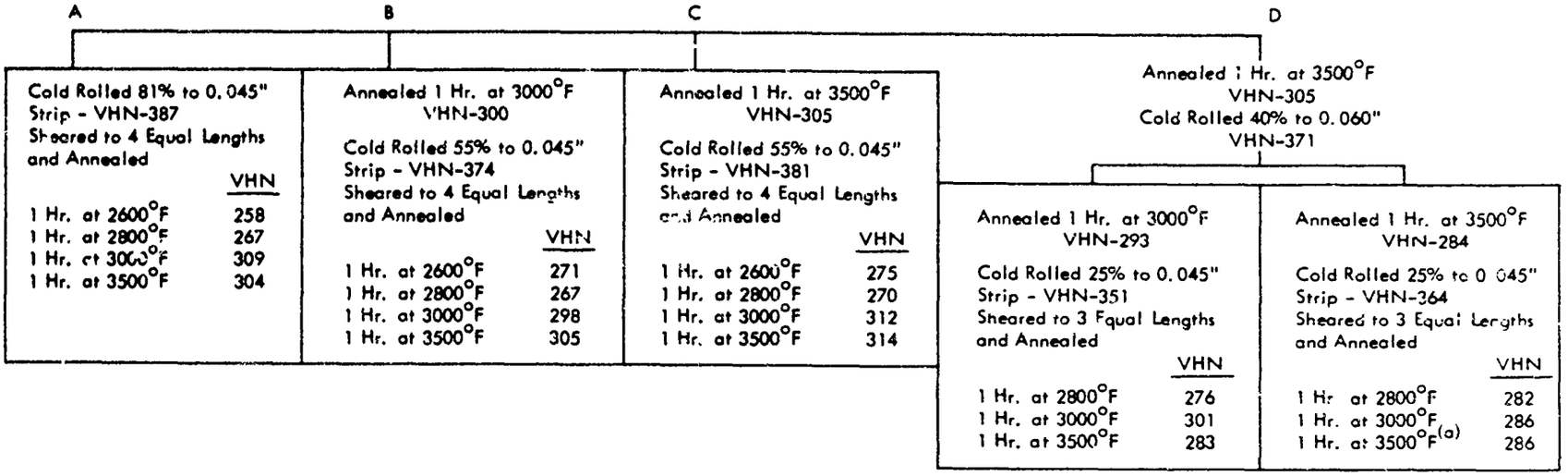
Tensile data obtained at 2400°F for the variously processed sheets are listed in Table 12 and shown graphically in Figure 25. The material, given a final anneal at 2600 and 2800°F, showed a large variation in properties due to the various stages of recrystallization represented. All of the material annealed at 3000°F was completely recrystallized and exhibited consistent strength properties. However, a trend toward lower elongation with higher intermediate annealing temperature was evident. The material annealed at 3500°F exhibited a variation in properties which appeared to be associated with the amount of prior reduction. The strength properties of the B and C material were comparable to the standard values while elongation values were slightly lower. The A, D1 and D2 material exhibited significantly lower properties in all respects. The D2 properties were substantially lower than those of the standard with an elongation of only 4%. (The grain size of all the 3500°F annealed material was in the ASTM-4 range). The carbon content of the D2 material was rechecked and found to be 0.0094% indicating little or no loss during the high temperature processing anneals. The room temperature hardness and elevated temperature tensile data indicated that the minimum temperature of 3000°F was required to completely recrystallize in 1 hour T-222 sheet which had been cold reduced 25 to 81%. The strength properties of the sheets annealed at 3000°F indicated no apparent influence of prior processing history with the possible exception of the D2 material. The ductility, however, showed a definite trend to lower values with increasing grain size. The yield strength of the D2 sheet was significantly higher while the elongation

Billet Ta-39-3 - 2-7/8" Dia. x 1-7/8"
Al-Si Coated - Upset Forged 71% at 2350°F
As-Forged Size - 5-3/4" Dia. x 0.536" - VHN-370

Conditioned to 3-1/2" x 3 1/2" x 0.445"
and Annealed 1 Hr. at 3000°F - VHN-311

Cold Rolled 46% to 6" x 3-3/4" x 0.240" - VHN-363
Annealed 1 Hr. at 3000°F - VHN-298

Cross Rolled (cold) 58% to 8-1/2" x 6-1/4" x 0.10" - VHN-379
Sheared to Strip



(a) Carbon content 0.0094% after final anneal.

FIGURE 24 - Mechanical-Thermal Processing of Heat Ta-39-3

TABLE 12 - Tensile Properties at 2400°F as Effected by Mechanical-Thermal History (Heat Ta-39-?)

Sheet* Identification	Ultimate Tensile Strength (ksi)	Yield Strength (0.2% Offset) (ksi)	Elongation (%)	Avg. Grain Diameter (mm)
A-2600	43.7	33.1	46	--
A-2800	41.7	32.3	58	--
A-3000	58.3	38.0	28	0.021
A-3000	58.7	37.9	27	--
A-3500	48.3	34.5	14	0.107
B-2600	58.0	42.8	29	--
B-2800	55.8	38.0	35	--
B-3000	58.0	36.8	24	0.025
B-3000	58.2	39.4	29	--
B-3500	57.8	35.9	21	0.097
C-2600	60.0	48.7	23	--
C-2800	46.8	33.0	43	--
C-3000	55.3	35.4	21	0.045
C-3000	58.0	39.3	25	--
C-3500	55.8	34.8	19	0.100
D1-2800	52.6	40.3	21	--
D1-3000	58.5	37.3	18	--
D1-3000	58.3	37.4	18	0.037
D1-3500	43.4	32.0	10	0.115
D2-2800	46.8	43.2	10	--
D2-3000	54.7	44.5	11	--
D2-3000	55.0	43.8	12	0.105
D2-3500	34.5	31.3	4	0.110

* Refer to Figure 22 for Mechanical-Thermal History

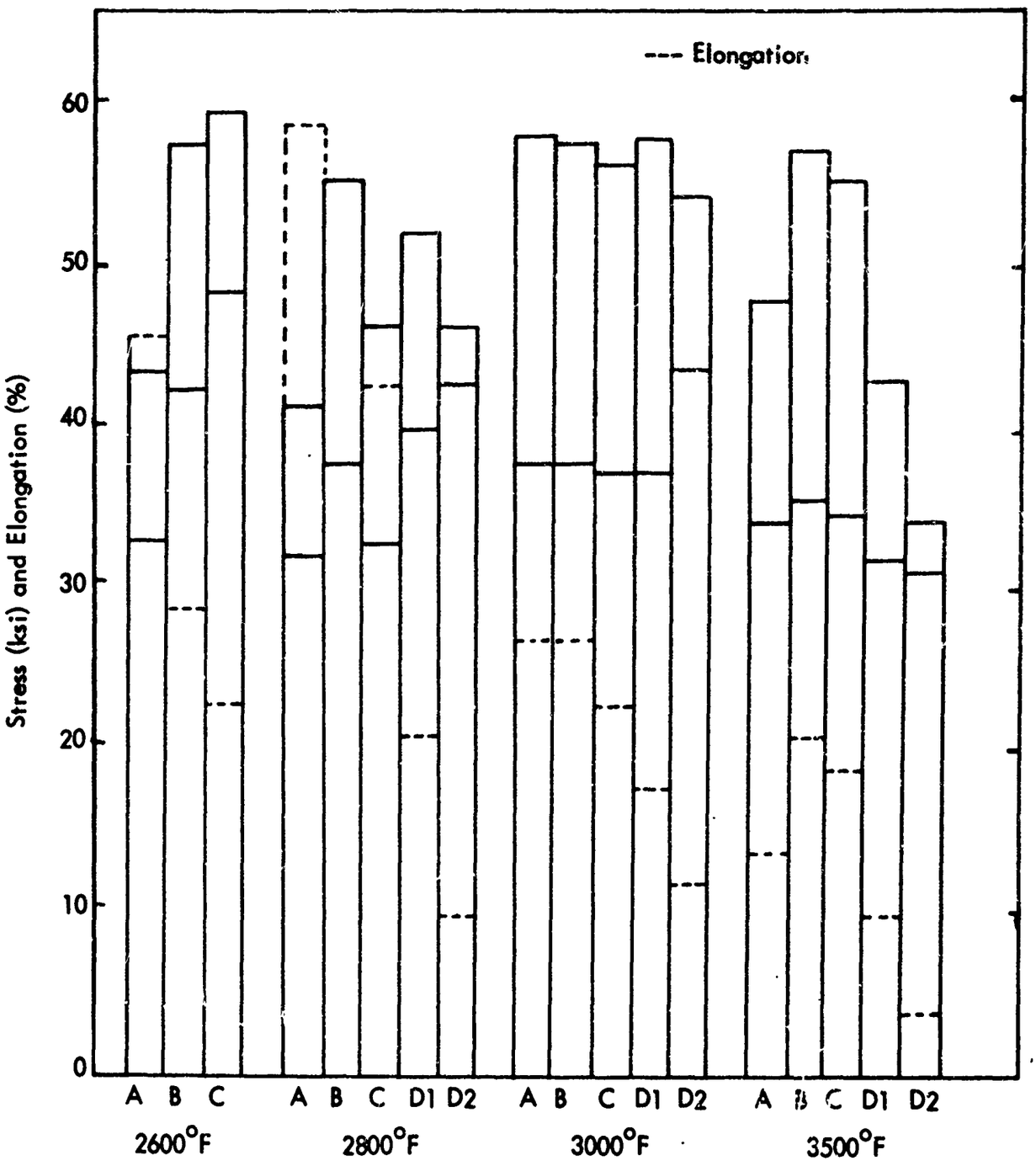


FIGURE 25 - Tensile Properties at 2400°F As a Function of Prior Thermal-Mechanical History for Heat Ta-39-3

was correspondingly lower than the other 3000°F annealed material. The high yield strength and low elongation may be associated with the larger grain size of the D2 material as compared to the other 3000°F annealed sheet.

It was also noted that the final grain size of the 3500°F annealed sheets was apparently independent of prior history. The final grain sizes of the sheets representing the various processing conditions were approximately the same.

Because of the varied stages of recrystallization present in the samples annealed at 2600 and 2800°F prior to testing at 2400°F, the influence exerted on mechanical properties by second phase precipitation could not be evaluated as was done for heat Ta-43. The necessity of a minimum 1 hour annealing temperature of 3000°F to insure consistent strength which, within rather broad limits is independent of prior history, coupled with the tendency toward loss of ductility resulting from higher temperature intermediate and/or final anneals further verifies the choice of the 3000°F final anneal treatment as optimum to produce the best combination of properties in T-222.

STRENGTHENING MECHANISMS

In the preceding contract periods, it was shown that C and N interstitial solute additions to the base Ta-W-Hf composition make a significant contribution to high temperature strength. This contribution to strength is a maximum at about 1800°F and persists up to about 3000°F. The mechanism whereby these interstitial solutes contribute to high temperature strength is not known. However, the experimental data discussed previously suggest that the mechanism may be that of immobilization of dislocations by precipitate pinning, as suggested by Chang[6] and Gregory.[7]

A plot of strength versus temperature shows a peak in ultimate strength occurs at about 1800°F, and this peak occurs in T-111 as well as in T-222. In the absence of any evidence for a temperature dependent ordering or clustering effect, this peak in strength is attributed to the

presence of interstitial solutes in the material. It was also previously shown that C and N levels of about 200 ppm which gave a single phase microstructure at the solution treating temperature were just as effective in promoting strength as higher levels of C and N (400-800 ppm) which gave a two phase microstructure at the recrystallization temperature. Thus, the strength contribution due to C and N appears to be related to the C and N initially taken into solid solution at the solution treating temperature.

Interstitial solutes in solid solution can contribute to strength, e. g., by segregating to dislocations, but this mechanism would not be expected to be very effective at high temperatures where the diffusivity of interstitials is high. Substitutional-interstitial solute interactions could lead to lower diffusivity, as well as clustering effects, but it seems more likely that actual precipitation is involved. However, it is unlikely that classical precipitation hardening is operative. The types of microstructure and precipitate morphology attained in T-222 alloy by aging the supersaturated solid solution were shown in Figures 20 and 21. The low volume fraction of precipitate and the coarse state of dispersion would seem to preclude the possibility of the precipitates effectively blocking the motion of mobile dislocations. This is borne out by the tensile test data described in Table 11, i. e., aging the solution treated material to produce the type of precipitate structures shown in Figures 20 and 21 inveriably resulted in a decrease in strength. Also, previous work[1,2] has shown from room temperature hardness measurements that T-222 alloy shows no response to aging.

In contrast to mobile dislocation-precipitate interactions in the usual sense of dislocation theories of precipitation hardening, strengthening due to precipitation on dislocations results from the decreased number of dislocations participating in the deformation process. In this case, the precipitates might well be too small to be observed except by electron transmission microscopy; thus the material would appear to be single phase as judged by optical metallography. It might also be noted that there is sufficient C or C+N present in T-222, and even T-111 to produce substantial pinning. In view of the strain induced precipitation of the FCC (Hf,Ta,W)C carbide observed in the phase identification work, it is probable that it is precipitation of this carbide on dislocations, during testing or during cooling from the solution anneal,

that is responsible for the observed strengthening effect due to interstitial solute additions to the base Ta-W-Hf alloy.

VII. CONCLUSIONS

1. The excellent combination of fabricability, strength, ductility and weldability previously displayed by T-222 was retained in alloy scale-up to 4 inch diameter ingot and production of 18 and 24 inch wide sheet.

2. Two carbide phases (M_2C and MC type) were observed on aging solution treated T-222 in the temperature range 1600 to 2800°F. The MC phase was stable at the higher temperature. Aging solution annealed plus cold worked material resulted in precipitation of only the MC phase.

3. In the recrystallized condition T-222 alloy was strongest when solution treated to provide a single phase microstructure. Aging solution treated material to produce second phase precipitation results in lower strength at both room and elevated temperatures.

4. Intermediate and/or final annealing treatments of T-222 alloy above 3000°F tend to result in loss of ductility in the final product, while the effect of prior history may not be eliminated by lower temperature anneals.

5. The best combination of strength and ductility for T-222 alloy, within broad limits, is obtained by 1 hour anneal at 3000°F.

VIII. REFERENCES

1. R. L. Ammon and R. T. Begley, "Pilot Production and Evaluation of Tantalum Alloy Sheet", Summary Phase Report, Bureau of Naval Weapons Contract NOw-62-0656-d, June 15, 1963.

2. R. L. Ammon and R. T. Begley, "Pilot Production and Evaluation of Tantalum Alloy Sheet", Summary Phase Report Part II, Bureau of Naval Weapons Contract N600(19)-59762, July 1, 1964.

3. Evaluation Test Methods for Refractory Sheet Materials, Report MAB-192M, April 22, 1963.

4. Metals Handbook, Vol. 1, Properties and Selection of Metals, 8th Edition.

5. E. Rudy, "Part II Ternary Systems, Vol. 1. Ta-Hf-C System Technical Report No. AFML-TR-65-2, Aerojet-General Corp., June, 1965.

6. W. H. Chang, "A Study of the Influence of Heat Treatment on Microstructure and Properties of Refractory Alloys", Technical Documentary Report No. ASD-TDR-62-211, Part III, August, 1964.

7. D. P. Gregory, "Mechanisms of Work Hardening in Body Centered Cubic Metals", ASD-TDR-62-354, Part III, March, 1965.

Astronuclear Laboratory

ACKNOWLEDGMENTS

The authors wish to acknowledge the contribution to this program of the following people.

Westinghouse Astronuclear Laboratory

Alloy Preparation	R. Bartifay
Heat Treatment	R. Fedorchak
Metal Working	R. Sprecace, C. Fitterer, R. Hough
Welding	G. G. Lessmann, R. Sprecace
Mechanical Testing	E. Vandergrift, J. Lesczynski
Metallography	K. Galbraith
Electron Microscopy	R. Goodspeed, W. Harding
X-ray Diffraction	A. Danko, R. Conlin

Westinghouse Research and Development Center

Chemical Analysis	O. Kriege, J. Rudolph

Astronuclear Laboratory

ACKNOWLEDGMENTS

The authors wish to acknowledge the contribution to this program of the following people.

Westinghouse Astronuclear Laboratory

Alloy Preparation	R. Bartifay
Heat Treatment	R. Fedorchak
Metal Working	R. Sprecace, C. Fitterer, R. Hough
Welding	G. G. Lessmann, R. Sprecace
Mechanical Testing	E. Vandergrift, J. Lesczynski
Metallography	K. Galbraith
Electron Microscopy	R. Goodspeed, W. Harding
X-ray Diffraction	A. Danko, R. Conlin

Westinghouse Research and Development Center

Chemical Analysis	O. Kriege, J. Rudolph

Astronuclear Laboratory

WANL-PR-M-014

October 30, 1965

ERRATA

Please substitute the attached sheet for Page 30 in the report "Pilot Production and Evaluation of Tantalum Alloy Sheet", Summary Phase Report Part III. The report was prepared under Contract NOw-64-0394-d, by R. L. Ammon, A. M. Filippi, and D. L. Harrod of the Westinghouse Astronuclear Laboratory.

Tensile properties reported erroneously for T-222 heats Ta-37-1 and Ta-37-3 have been corrected.

TABLE 6 - Comparison* of Elevated Temperature Tensile Results for Several T-222 Heats (Continued)

Identification	Ultimate Tensile Strength (ksi)	Yield Strength 0.2% Offset (ksi)	% Elongation (in 1 Inch)
	Test Temperature 3500°F		
Ta-37-1	15.9	15.8	37
Ta-37-2	14.1	14.0	43
Ta-37-3	12.6	11.6	12
Ta-39-1	14.2	--	70
Ta-39-1	13.3	12.8	83
Ta-40	13.7	13.3	48
Ta-43	14.3	14.3	45
Ta-43	15.5	15.5	38
	Stress Relieved 1 Hour at 2000°F - Test Temperature 1800°F		
Ta-37-1	100	89.2	10
Ta-37-3	101	87.2	8
Ta-43	102	90.7	6
Ta-43	102	87.7	6
	Test Temperature 2200°F		
Ta-37-1	78.8	68.8	18
Ta-37-3	91.8	81.8	14
Ta-43	77.7	62.9	21
Ta-43	77.8	69.2	15
	Test Temperature 2400°F		
Ta-37-1	61.3	56.0	24
Ta-37-3	55.2	45.6	31
Ta-43	61.8	47.5	25
Ta-43	58.7	49.5	30

* Results for Heats Ta-37, 39, and 40 obtained from Reference 2. All samples represent the longitudinal test direction.